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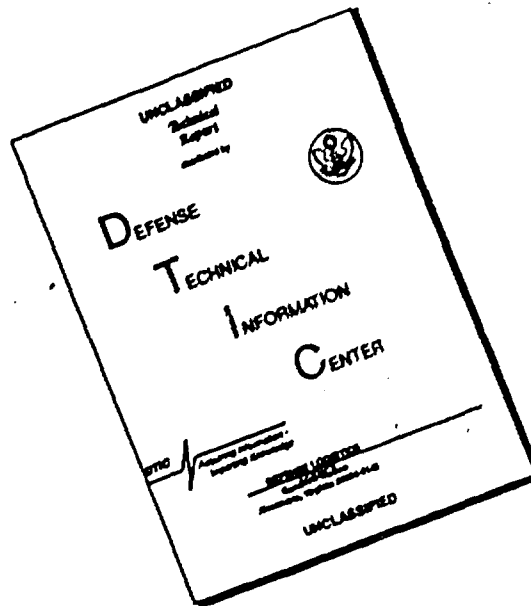
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# AZUSA PLANT

STRUCTURAL MATERIALS DIVISION

## DEVELOPMENT OF IMPROVED
## HIGH-STRENGTH PREIMPREGNATED
## MATERIALS FOR FILAMENT-WINDING

A REPORT TO

### BUREAU OF NAVAL WEAPONS

CONTRACT NOw 61-0642-C (FBM)

REPORT NO. 2577 / JUNE 1963 / COPY NO.

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**AEROJET-GENERAL CORPORATION**

AZUSA, CALIFORNIA

**AEROJET**

**GENERAL TIRE**

**GENERAL**

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## PREIMPREGNATED MATERIALS FOR FILAMENT WINDING

a report to

BUREAU OF NAVAL WEAPONS

Washington, D C.

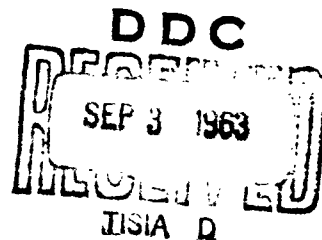Contract NOw 61-0642-c(FBM)

Report No. 2577                                                    June 1963

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# AEROJET-GENERAL CORPORATION
**A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY**

## FOREWORD

This is a summary report of the activities of the Aerojet-General Corporation, covering the period of 26 June 1961 through 1 February 1963, on Contract Number NOw 61-0642-c(FBM). This contract is under the direct supervision of the Special Projects Office of the Bureau of Naval Weapons, with H. Bernstein acting as technical monitor.

This program is being conducted by the Materials Engineering Department of the Structural Materials Division, Aerojet-General Corporation, Azusa. Major responsibility for the program resides with Ira Petker. Other significant contributors to the program include Dr. S. Brelant and M. Segimoto.

AEROJET-GENERAL CORPORATION

Dr. S. Brelant, Head
Materials Engineering Dept.
Structural Materials Division

## ABSTRACT

This program was a study of the processes used in the manufacture of 20-end ECG fibrous-glass roving and preimpregnated roving made from it. The program was composed of two independent phases. Phase I was a critical analysis of the various steps involved in the manufacture of glass roving from the point of drawing glass filaments to packaging and shipment of the product. The purpose was to establish optimum procedures which would lead to a retention of a greater proportion of the fiber's "virgin" strength. Test results for eight experimental lots of roving produced for this study are presented. Data includes gravimetric measurements and tensile strength of the AeroROVE strands, NOL rings, and 18-in.-dia chamber for both dry and preimpregnated rovings. Production preimpregnated rovings in which were incorporated improvements developed during this program have shown a tensile strength increase of approximately 25%. Also, improvements resulting in more uniform lineal weight and sizing content of roving have been achieved.

Phase II was a prepreg manufacturing process variable study. The purpose of this study was to obtain a clearer and more quantitative understanding of the effects of preimpregnation manufacturing parameters on the properties of prepreg roving. Sixteen experimental runs were made to investigate seven process variables, and two runs were made to improve and control resin content in prepregs by application of specially designed devices. Inspection test data for all these material lots are presented, as well as specific process conditions used to produce these lots. These data include gravimetric measurements (volatile, resin content, and weight per linear yard), resin flow, gel time, viscosity index, and tensile strength (both AeroROVE strands and NOL rings). A viscosity-index test was developed to measure the degree of resin polymerization and applied successfully to the analysis of the effects and the sensitivity of prepreg to variations in the parameters of preimpregnating processing. An approach to setting quantitative control limits in the manufacture of prepreg has been demonstrated by the application of the viscosity-index test as a means of correlating differences in processing characteristics of prepreg.

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APPENDIX B - PROCEDURE FOR THE DETERMINATION OF GEL TIME

APPENDIX C - PROCEDURE FOR THE BAND WIDTH MEASUREMENT

I.   INTRODUCTION

This program was a joint effort of Owens-Corning Fiberglas Corp., U.S.
Polymeric Chemicals, Inc., and Aerojet-General Corp.  The purpose of the program
was to develop an improved, preimpregnated glass-fiber material (prepreg) suit-
able for use in filament-wound motor cases capable of withstanding tensile strength
(fiber-stress) levels of from 375,000 to 400,000 psi.

The original program was to cover an 8-month period and was to be divided
into two phases.  Phase I was to establish optimum procedures for fabrication,
handling, and shipping glass roving and for applying controlled quantities of
resin to glass-roving materials.  Specific tasks on Phase I were:  (a) fabrica-
tion of eight 100-lb lots of E-glass with HTS sizing by Owens-Corning under con-
trolled, documented conditions;  (b) determination by Aerojet of the mechanical
properties of each lot of glass; (c) impregnation by U.S. Polymeric of each lot
of glass under controlled, documented conditions; (d) evaluation of each lot of
prepreg by Aerojet; and (e) preparation of tentative material and process speci-
fications for high-strength, high-quality prepreg.  Phase II was to be a confir-
mation of the positive results of Phase I with a larger sampling of material pre-
pared under production conditions.

Improvements in strength of production glass roving by incorporating
changes which were developed for the first few lots of this program negated the
need for Phase II as originally planned.  Phase II was changed to a critical
study of the preimpregnation process and its effect upon the processing character-
istics of prepreg.  Since Phases I and II in the revised program were essentially
independent studies, for the sake of clarity they will be treated separately in
this report.

## II.    PHASE I - STUDY OF ROVING FORMING PROCESS

### A.    DESCRIPTION OF STUDY

In filament winding, the basic type of reinforcement currently in use is unwoven glass filament in the form of multiple-end roving. Figure 1 is a schematic rendering of the process which is used to produce roving and includes all the basic operations applied to the glass fiber prior to its impregnation with a resin. The important steps in the process shown in Figure 1 are as follows:

1.    Blending of raw materials used in the glass (A)

2.    Melting of the glass raw materials (B)

3.    Formation of the glass marbles (C)

4.    Remelting of glass marbles in a bushing (D)

5.    Gravity extrusion of glass through orifices in the bushing and formation of "virgin" fibers (E)

6.    Application of size or finish to fibers (F)

7.    Collection of fibers into a single end (G)

8.    Winding of single end onto forming tube or cake package (H)

9.    Oven bakeout of forming tube to remove excess volatiles from sizing (I)

10.    Placement of a group of forming tubes on a creel (J)

11.    Collection of a group of single ends through a guide eye to form a roving (K)

12.    Windup of the roving to form a roving spool (L)

13.    Packaging and shipment of roving (M).

Just after Step 5 (Figure 1,E) fibers formed from E-type glass composition have been reported by numerous observers to have strength in excess of 500,000 psi. A short time ago only 50% of this strength, or 250,000 psi, was retained when multiple-end roving was tested after Step 13. The purpose of

II Phase I - Study of Roving Forming Process, A (cont.)

Phase I of this program was to analyze critically all steps in the roving forming process between Steps 5 and 13 for their contribution to this loss in strength. New methods were then to be developed and applied to those points in the process which would lead to a retention of a greater proportion of the original strength of the fibers.

The approach taken to this problem involved the preparation of experimental lots of roving by Owens-Corning. Each experimental lot incorporated certain potential improvements and consisted of ten 10-lb rolls of 20-end roving. Each lot was 100% inspected by Owens-Corning, using the following tests.[*]

| | |
|---|---|
| lay | strand yardage |
| waywind | roving yardage |
| package length | strand moisture |
| diameter | strand volatiles |
| roving weight, net | strand solids |
| centering on tube | roving moisture |
| strand width | roving volatiles |
| strand spacing | roving solids |
| stops/doff | wetout rate |
| fuzz content | laminate clarity |
| stiffness | shelling |
| ribbonization | catenary |
| end count | durometer hardness |
| | tensile strength |

Each experimental lot of roving was shipped to Aerojet, Structural Materials Division, where the following tests were performed: (1) vinyl strand strength, (2) 1/16-in. NOL-ring strength, (3) ignition loss, (4) weight per lineal yard, and (5) 18-in.-dia chamber strength.

The roving was then preimpregnated by U.S. Polymeric with E-787 type resin under constant impregnating conditions. It was then tested again by Aerojet for possible strength losses due to preimpregnation. The prepreg tests applied by Aerojet were the following: (1) prepreg strand strength, (2) 1/6-in. NOL-ring strength, (3) volatile content, (4) resin content, and (5) 18-in.-dia chamber strength.

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[*] Owens-Corning Fiberglas Corp. Customer Acceptance Standards TP-245, 21 September 1961.

II Phase I - Study of Roving Forming Process, A (cont.)

All significant changes leading to improved strength or quality in the roving which were noted during the course of this program were to be incorporated into the standard production process by Owens-Corning.

B.    DESCRIPTION OF EXPERIMENTAL LOTS OF ROVING

1.    Lot 1 - Improved Package

a.    More uniform tension was applied to each individual strand (end) through the use of more efficient guide eyes, alignment, and positioning.  Automatic knock-off devices were maintained in the thread line to aid in assuring absolute end count (Step 11)[*].

b.    Incorporation of an "interlocking waywind." This refers to the internal characteristic of the roving package in the sense that each successive band (wrap) tends to lock preceding bands in place.  Its use is to give greater stability to the roving package and to prevent sloughing (Step 12).

c.    Development of a flatter band.  This was accomplished through use of newer techniques in the areas of winding equipment, guides, and roving-package tensioning (Step 12).

d.    Use of a stronger roving ball core.  The need for greater strength was to preclude collapsing of the ball after preparation and during shipping (Steps 12 and 13).

e.    End caps were employed to protect the prepared ball during handling, shipping, and storage.  The core was changed for the same basic reason to a longer tube with a knurled surface to help prevent slipping of the ball on the core after production and to allow for end pads and caps (Step 13).

f.    The roving was sealed in a polyethylene bag to protect it from contamination by moisture and foreign matter and to impress upon production personnel the "special" nature of the product being manufactured (Step 13).

g.    The package hardness characteristics were improved by tightening the winding specifications to achieve more consistent measurements and

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[*]Numbers in parentheses in Section II,B refer to the respective processing steps described in Section II,A.

II Phase I - Study of Roving Forming Process, B (cont.)

and setting minimum hardness limits in order to achieve better product uniformity (Step 12).

        h.    A larger and stronger shipping carton was developed to provide necessary space to package the roving, including changes made in d, e, and f above, and for additional protection to the product during shipping and warehousing (Step 13).

        i.    A new palletizing method was used consisting of three layers per pallet (24 cartons per layer) with corrugated layer caps over each layer to interlock the load. Damage-free freight cars were used to permit separation of a carload lot by means of cross bars in such a manner as to preclude shifting of the entire load in transit and to guard against other damage (Step 13).

        2.    **Lot 2 - More Uniform Surface Treatment (Step 10)**

        a.    Only a portion of each forming tube was used based on sizing migration curves. That portion where lowest variation was measured was used to prepare this lot of roving.

        b.    Distribution or migration curves of variation of sizing content within forming cake packages have been developed for HTS forming stock.[*] Various degrees of migration are measurable in many types of forming stock, including HTS.

        3.    **Lot 3 - Improved Strand and Roving Weight Variation**

        a.    Input was selected for uniformity in strand coating plus better-than-standard control of bare glass yards per pound (Steps 5 and 6).

        b.    Only two balls of roving were prepared from each set of 20 pre-tested tubes to minimize variation in weight per yard due to possible migration effects. This method was used in an attempt to asure a minimum variation in weight per yard throughout the sample roving packages.

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[*]Additional details considered proprietary information by Owens-Corning and therefore not supplied by them.

II Phase I - Study of Roving Forming Process, B (cont.)

4.    Lot 4 - Roving Stiffness Variation (Step 9)

Several methods of increasing the stiffness of HTS roving were investigated.  The method which was selected yielded the most consistent results with a minimum difference in other characteristics or measurable properties. Additional heat application was the approach taken.*

5.    Lot 5 - Package Moisture Variation

The roving was exposed to high humidity conditions for a period of time and rechecked for evidence of moisture penetration.  Testing by Owens-Corning revealed moisture introduction at a level of 0.1% or less.  Re-testing of significant properties exhibited lower degree of wetout.  (The conclusion of Owens-Corning was that a meaningful amount of moisture had been introduced into interstices of the roving package.  However, it was their contention that the strand coating had not been penetrated to any significant degree, if at all.)

6.    Lot 6 - Catenary Improvement, Parallel-Wound Package

a.    According to Owens-Corning, the best thinking of Owens-Corning and winding equipment manufacturers was combined in several attempts to reduce catenary, but they were unable to improve upon the available package supplied for Lot 1.

b.    Pre-tested stock was prepared to be fabricated into roving by the Ashton Product Development Laboratory (OCF).  After preparation four packages were sent to the Anderson plant for testing.  Results did not indicate an improvement in catenary reduction.  Laboratory personnel believe that the nature of a helical-wound package was the major reason for their failure to accomplish catenary reduction.

c.    It was decided to use a level-wound package for this lot.  The level-wound or parallel-wound package was, in the estimation of Aerojet and Owens-Corning, the best way to proceed.  Essentially, parallel wraps were placed on a double-flanged spool with spacing between successive wraps.  An

*Additional details considered proprietary information by Owens-Corning and therefore not supplied by them.

II Phase I - Study of Roving Forming Process, B (cont.)

available smaller-capacity spool was employed (Step 12).

### 7. Lot 7 - Finer Filament Input Stock (Step 5)

DE filaments (approximately 408 filaments per end) were used in place of the standard G filaments (204 filaments per end).

### 8. Lot 8 - Zero Ribbonization

Methods employed were a combination of heat and chemical differences. The roving was otherwise made as nearly as possible in conformance with OCF CAS TP-245 (Step 9).

### C. PREIMPREGNATION

In this program glass roving, after testing by Aerojet, was sent to U.S. Polymeric, where it was coated with E-787 type resin. Figure 2 is a schematic diagram of a typical impregnation process used to produce prepreg. The main steps in the preimpregnation process are as follows: (1) positioning of the roving spools on a creel, (2) coating of roving with resin by passing roving through a diluted solution of resin, (3) removal of solvent and partial polymerization of resin by means of heat within the drying tower, and (4) windup of B-staged prepreg.

For the preimpregnation of the experimental lots of roving, no deviations were allowed in the impregnation process except as required to control resin content. The important processing parameters and their levels were:

| Parameter | Average Level |
|---|---|
| Tower temperature | $360 \pm 10^{\circ}F$ |
| Processing speed | $61 \pm 10$ ft/min |
| Creel tension | 750 g |
| Resin-gel time | $4 \pm 0.25$ min |

### D. TEST RESULTS

Test data for all materials is summarized in Table 1 for dry glass and in Table 2 for prepreg. Data from 18-in. chambers is summarized in Table 3. As shown in Table 1, the average lot strength of vinyl-coated strands for all

II Phase I - Study of Roving Forming Process, D (cont.)

lots was between 302,000 and 348,000 psi. Five of the eight lots, Lots 1, 3, 4, 5 and 7, had strand strength within a much narrower range, from 335,000 to 348,000 psi. The two lots with the lowest strength, about 303,000 psi, had low ribbonization. The average lot NOL-ring fiber strength was between 319,000 and 349,000 psi for the same five lots which had high strand strength. Lot 2 and the lots with low ribbonization (Lots 6 and 8) had NOL-ring fiber strengths of about 303,000 psi. Sizing content, as measured by ignition loss, tended to be quite consistent with a nominal average for all lots of about 1.45%. The weight per yard was also quite consistent at about 0.652 g.

As shown in Table 2, the lot average range for prepreg strands was between 292,000 and 340,000 psi. In general, the prepreg strand values were lower than the vinyl strand values for the same lots. However, NOL rings were very consistent between lots and also tended to be stronger than the equivalent in-process rings. With the exception of Lots 3 and 6, the lot average range for prepreg NOL rings was 335,000 to 352,000 psi. In most instances, the prepreg NOL rings failed at higher stress values than vinyl-coated strands from the same lot. A particularly large difference of this type was noted for Lot 8, in which the average of 304,000 psi for vinyl strands is considerably lower than the figure of 352,000 psi for prepreg NOL rings. The prepreg gravimetric data tends to vary somewhat more than is desirable, although in respect to resin content, Lot 7 (DE filaments) had an unusually small total variation between rolls (1%).

The data from the 18-in.-chamber tests, summarized in Table 3, does not indicate any consistent trend. Several chambers, particularly those prepared from Lots 3 and 5 prepreg, had hoop-filament stresses greater than 330,000 psi. However, other chambers had hoop filament stresses as low as 260,000 psi.

E.    TECHNICAL DISCUSSION

1.    Glass Strength

Certain inconsistencies appear in the test data when the data between dry glass and prepreg material, and from one test to another is compared. The prepreg strand strength is generally lower than the vinyl-coated strand strength, whereas the prepreg NOL-ring strength is higher than in-process NOL

II Phase I - Study of Roving Forming Process, E (cont.)

ring strength. It was also found that the vinyl strand strength was higher in general than in-process NOL-ring strength. However, prepreg strand is consistently lower than prepreg NOL ring strength. Finally, for certain lots, including Lots 2, 4, 5, 6, 7 and 8, the prepreg NOL-ring strength is equal to or higher than the vinyl strand strength measured for the same glass prior to preimpregnation. Since strand tests, NOL-ring tests, and 18-in.-chamber tests are the criteria used to evaluate improvements in the prepreg, a discussion and interpretation of this data at this point appears warranted.

The strand test is essentially a test of pure tension, whereas NOL ring testing, by its nature, imposes bending and shear forces which will cause glass failure at a lower stress level than the same glass fibers in pure tension. If the hypotheses are made that the glass fibers are strongest when stressed in pure tension, and that the preimpregnation process may or may not lower, but will not improve, the glass strength, the data can be interpreted more meaningfully. First, the highest glass strengths within any given lot of roving should have been for the vinyl strands; these values should be higher than the corresponding NOL-ring strengths. Since this was not true in several instances, it can be concluded that the vinyl strand test does not stress the glass to its ultimate capability; actual strength of the glass, therefore, is probably higher than is indicated by the test data, and the strand test data should be viewed as defining minimum rather than ultimate strength. The inability of the vinyl strand test to approach ultimate glass capabilities is associated with the properties of the vinyl resin and with the methods of gripping. A resin which has better wetting characteristics, higher shear strength, and more efficient shear transfer characteristics than the vinyl resin employed (i.e., the same type of resin used in chamber fabrication) would probably produce higher calculated glass stresses. Also, an improved method for distributing the load in the grip area more uniformly would also reduce borderline grip failures.[*]

The results obtained with prepreg strands are consistently lower than those obtained with prepreg NOL rings, indicating again that problems

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[*] Recent work in the Structural Materials Division has tended to verify these conclusions. Further indications have been that all strand tests currently in use suffer from these same problems.

II Phase I - Study of Roving Forming Process, E (cont.)

associated with the test methods have introduced some complication to the
simple analysis of the test data.  However, the lower strength of the prepreg
strand, compared to the vinyl strand, still requires an explanation, since pre-
preg NOL rings and 18-in.-chambers have consistently been equivalent to the higher
end-strength and in-process NOL rings and chambers made with the same glass.
It is particularly important to note that prepreg strand data is the result of
a test of a composite rather than a simple test of glass strength.  Although the
amount of resin present in prepreg strands is theoretically great enough to com-
pletely impregnate the fibers, there is little excess resin available to make up
for localized resin deficiencies, or to prevent sequential failure and peeling
at localized fiber breaks.  In addition to the low resin content, no pressure
is used in preparing specimens; therefore, little effective resin migration takes
place, which could compensate for local resin inadequacies and which could pro-
duce more uniform impregnation.  Finally, in the grip area, little excess resin
is available to protect the fibers and distribute the loads created by the fric-
tion grips.  The sum total of these factors can make prepreg strands very sensi-
tive to minor variations in resin distribution, spatial relations between ends,
band width, position of filament flaws, etc.  Indeed, roving impregnated in the
laboratory, using the same resin systems, has yielded prepreg strand data which
is equivalent to or higher than the vinyl strands.

Some preliminary work has been done with vinyl-coated prepreg
strands, the results of which are shown in Table 4.  All these strands were pre-
pared in the same manner as standard prepreg strands, except that the prepreg
was coated with vinyl resin prior to oven cure.  In each case, the vinyl coating
increased the failure stress significantly; the most dramatic effect was noted
on the strands which orginally failed at the lower stress levels.  This data
indicates that improvements in strand grips which tend to distribute gripping
loads more satisfactorily will lead to higher measured stress values.

Another interesting comparison was made between prepreg and
in-process NOL rings; it was found that the prepreg rings were consistently higher
in strength than in-process rings for the same lots of glass.  Based on the
original hypothesis that preimpregnation cannot improve glass strength, the

II Phase I - Study of Roving Forming Process, E (cont.)

explanation for the strength improvement must be found in factors other than glass strength. Although no quantitative analysis is possible at this time, it would appear that the differences would be related to composite differences such as resin content, void content, interfacial shear, etc., or to resin differences such as shear strength, wettability, rigidity, etc. Since ring testing appears to be sensitive to one or more of these factors and does not measure a true physical property characteristic of any individual component of the composite, caution must be exercised in attributing differences in NOL-ring strength to differences in the glass strength.

2.    Gravimetric Data

a.    Roving

Ignition loss (or sizing content) for all experimental lots of material, except Lot 2, has been very consistent both between lots and between rolls within a given lot. (Lot 7 is an exception but this is probably because Lot 7 represented the first DE filaments treated with HTS-type finish.) This improvement is due, at least in part, to the practice of discarding a portion of the roving from the inside and outside of each cake package in which sizing content is most variable. The variability at these positions is due in part to migration effects; these effects occur while the size is liquid, prior to and during the oven bakeout cycle of the cake packages. Volatile losses are probably the highest on the outside of the cake packages, since that surface is in direct contact with heated air during the oven cycle.

As with ignition loss, weight per lineal yard has been very consistent for all lots of roving. The maximum weight range within any given experimental lot has been 0.015 g. This represents a 50% improvement over the previous HTS-E roving, for which a range of over 0.030 g was usually measured.

b.    Prepreg

For the impregnation of all experimental lots of material, no deviation was allowed in the basic impregnation process except as required to maintain a uniform resin content. The main impregnation processing parameters and their average levels were:

II Phase I - Study of Roving Forming Process, E (cont.)

| Parameter | Average Level |
|-----------|---------------|
| Tower temperature | $360 \pm 10°F$ |
| Processing speed | $65 \pm 10$ ft/min |
| Creel tension | $750$ g |
| Resin-gel time | $4 \pm 0.25$ min |

    The main parameters which did require adjustment between lots were the specific gravity of the resin bath and creel tension. Resin-bath specific gravity is the most influential factor in the determination of prepreg resin content. A change of 0.005 units of specific gravity causes a 1.0% change in resin content for 20 E HTS roving, with a ribbonization of 2 or 3, assuming otherwise constant resin pickup characteristics for the roving. The influence of creel tension can be explained by reference to Figure 2, which is a schematic diagram of a typical preimpregnation system. As shown in Figure 2, there are only two contact points at A and B prior to the oven. At E and F, work is applied to the roving, and the strand will tend to spread and break down into individual ends. Roving surface area will become larger as the number of unbonded ends becomes larger, and, other things being equal, the resin content will also become greater, since it is dependent upon the surface area of roving exposed to the resin in the impregnation bath. Therefore, resin pickup will vary directly with the amount of debonding that occurs at points A and B. The effect of creel tension is that, as it is raised, a greater amount of work will be applied to the roving at these contact points, and, therefore, a greater amount of debonding can occur. To sum up, resin content will be dependent on roving surface area at time of impregnation; this, in turn, is a function of degree of end-to-end bond, number, and type of contact points and creel tension.

    U.S. Polymeric has indicated that variability in end-to-end bond is the most important single roving characteristic responsible for variability in resin content. End-to-end bond may be defined as the amount of work required to break a single 20-end strand of roving into 20 single-end strands. If this property is variable, it is possible that during preimpregnation the degree of end-to-end bond breakdown will also be variable. As noted previously, little work is applied to the roving in the U.S. Polymeric process. The basis

II Phase I - Study of Roving Forming Process, E (cont.)

for eliminating work is the assumption that under these conditions the minimum amount of damage will be done to the roving. However, since it is impossible to eliminate all work from the process, debonding does occur for weakly bonded roving. It does not occur as much for strongly bonded roving. At present there is no method for measuring end-to-end bond and therefore no quantitative analysis of the effects of this property is possible. (The Owens-Corning ribbonization measurement does not consider end-to-end bond, since no method is incorporated in the test to apply a controlled and reproducible amount of work to the roving.)

Zero-ribbonization roving has been suggested as one means of eliminating the end-to-end bond problem since this type of roving, having no end-to-end bond, would have a controllable and reproducible surface area. However, it is important to note that any degree of ribbonization is acceptable from the standpoint of resin content uniformity if both the end-to-end bond and the work applied during the impregnation process are both controllable and reproducible. Experience with several preimpregnators who have used zero-ribbonization roving has indicated that the potential improvment in resin content control with this roving may be superseded by other less desirable characteristics. It appears that there is a tendency for catenary and twist buildup to occur during impregnation or zero-ribbonization roving. Normally, these effects are resisted by the end-to-end bond in standard roving. It has been reported that there is a rather high degree of twist and cross-over in zero-ribbonization roving and an apparent lack of it in standard roving. Since the roving forming process is the same for both materials, it would appear that the end-to-end bond in standard roving simply covers up these defects rather than being defect-free, as is normally assumed. However, if a zero ribbonization roving absolutely free of twist and crossover could be supplied, it would result in a prepreg with improved band width and resin-content control.

An alternative to zero-ribbonization roving would be to apply sufficient work to the roving during impregnation to overcome the maximum degree of end-to-end bond that might exist in the input roving. The potential damage to the roving would have to be considered in any attempt to solve the problem in this manner. However, both this approach, as well as zero-ribbonization

II Phase I - Study of Roving Forming Process, E (cont.)

roving, are being investigated in the current program.

Thus far, two parameters which affect resin content have been discussed. One factor, specific gravity, is related to the preimpregnation process; the other factor, end-to-end bond, is related basically to the roving but is also affected by the preimpregnation process. Two other roving properties, which affect resin content and which can be treated quantitatively, are weight per yard and sizing content. However, as will be shown, these properties explain only a small part of the resin content variation of prepreg. If a constant resin pickup is assumed, a variation of $\pm 0.03$ g ($\pm 5\%$)/yd of roving can account for a resin content variation of about $\pm 0.6\%$. However, as was noted previously, the total range of weight per yard in most of the experimental lots of roving was only 0.015 g, whereas the range in resin content was 0.9% for the best lot (Lot 7) and between 2 and 3.5% for all the other lots. It should also be noted that extreme care could be exercised in the impregnation of the experimental roving since only one or two rolls of roving were coated at any one time, compared to as many as 50 rolls under standard production conditions.

The contributions of sizing content to resin-content variation is even smaller, since a variation of $\pm 0.5\%$ in sizing content can cause a variation of only $\pm 0.06\%$ in resin content. Actually Lot 7, which had the lowest resin content variation, had one of the highest sizing-content variations of all the experimental lots of roving. Therefore, quantity of sizing and its variability is not a factor of major importance.

Other roving properties which may affect resin content are twist and crossover, affinity of the resin to the finish (wettability), degree of bond between filaments within an end, and degree of penetration of the finish by the resin. There may be other properties which might even be more important than any of those suggested; however, the important factor for all the properties in this group is that no quantitative value can be assigned to any of them at this time, and therefore no quantitative analysis is possible. That current quality control at Owens-Corning does not measure a property directly related to resin pickup characteristic is indicated by the data in Table 5; this table shows the lot averages, the average mean deviation, and the high and low value for the

II Phase I - Study of Roving Forming Process, E (cont.)

standard quality-control tests that are currently applied by Owens-Corning to
20 E HTS roving. This data is for the experimental roving supplied for this pro-
gram. At least one measurement of each property was made for every roll of
roving. In addition to these properties, measurements of catenary, ribbonization,
and yardage per pound were also made by Owens-Corning. Catenary and ribbonization
data are not included in Table 5 because Owens-Corning data indicated that the
former property was invariant at a level of zero and that the latter property
was invariant at a level of 2 or 3. (Yardage per pound is essentially the same
as weight per yard, which was discussed previously.)

Although several of the properties shown in Table 5
may be eliminated a priori as being related to resin pickup, such properties as
wetout rate, stiffness, solids content, and volatile content could conceivably
affect prepreg resin content. However, when these properties are compared to
the prepreg resin content data also shown in Table 5, no correlation or trend is
apparent.

c.      Chamber Data

Although in general the hoop filament stress at burst
of the 18-in.-chambers has been relatively high, few of the chambers have du-
plicated the high glass stresses of the simple tensile tests. In the fabrication
of an 18-in.-chamber, there are a number of variables which do not have a major
effect on simple tensile testing but which can affect chamber performance.
Filament alignment, winding pattern, tension uniformity, resin flow, air occlu-
sion, and mandrel contour are just a sampling of the parameters which can ad-
versely affect chamber burst strength. Theoretically the strength in pure tension
of the input roving should be the maximum burst stress that can be expected of
a chamber, and the difference between this value and the filament stress at burst
should indicate the efficiency of the fabrication process. In Figures 3 and 4,
chamber filament stress at failure is shown in terms of efficiency factors based
on input glass strength. In Figure 3, the efficiency factor was obtained by
dividing ultimate hoop filament stresses by lot-average NOL-ring strength. In
Figure 4 the efficiency factor used is ultimate hoop filament stress divided by
Aerorove strand strength. It is apparent from Figures 3 and 4 that over 80% of

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the chambers failed at efficiency factors of 0.85 or higher. Although these
figures do not indicate a perfect correlation between ultimate chamber filament
stress and either strand or NOL-ring strength, it does point out those chambers
for which a serious processing deviation probably occurred. In a positive sense,
the figures also indicate that at least 85% of the NOL-ring or strand strength
can be expected in a biaxially loaded chamber.

    F.    CONCLUSIONS

        The study to date has produced the following significant conclusions:

        1.    The Lot 1 improved package has resulted in a higher average
dry-glass strength. This conclusion is substantiated by the strength of current
production prepreg. As shown in Table 6, the average lot strength of production
prepreg for the period between June 1962 and September 1962 was between 367,000
and 400,000 psi. Just prior to this period the average lot strength was about
340,000 psi. These values indicate a significant upward trend, especially when
compared to an average prepreg strand strength of between 260,000 and 300,000 psi
for production prepreg received during early 1962. The original contention of
this program that improvements to the roving forming process and more intensive
quality-control procedures would recover a major fraction of the virgin-glass
strength appears to be validated by the strength of current production roving.

        2.    The preimpregnation process does not materially damage roving,
but it does result in fabrication material which produces composites equivalent
to or better than those produced by the in-process or wet-impregnation process.

        3.    Current methods for measuring the tensile strength of glass
in a composite require modification and optimization before truly quantitative
analysis can be made of the efficiency of chamber processing, design, and the
influence of resin properties.

        4.    A method for measuring the resin pickup characteristics of
roving is highly desirable in order to guide the development of prepreg with im-
proved resin-content control and wetout characteristics. There are several roving
properties of importance in this regard, and probably each should be treated
independently. Of major importance would be methods to measure end-to-end bond,

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twist-and-crossover, and the variability of finish to wetting.

5.     Zero-ribbonization roving would probably permit improved control of prepreg resin content and bond width.  However, to be used successfully, twist and crossover would probably have to be eliminated from the roving and careful control would be necessary during impregnation in order not to introduce fiber misalignment.  An alternative approach would be to form prepreg directly from single end strand, utilizing dies to form a band of uniform thickness and width.

6.     Preliminary work with DE filaments has indicated a potential improvement to be obtained from their use.  Although there appears to be no strength advantage in filaments of smaller diameter, resin-content control, and composite uniformity may be improved.

III.    PHASE II - PREIMPREGNATION VARIABLE STUDY

A.    PROGRAM OUTLINE AND TEST PROCEDURES

The object of the revised Phase II program was to obtain a clearer and more quantitative understanding of the effects of preimpregnation processing parameters on the properties of prepreg roving.  The program was divided into four main subtasks, each containing several related parts.  These subtasks were process-variable study, resin-content study, band-width study, and processability upon aging.  The types of information to be obtained from the work were the following:  (1) Definition of the variation of resin content and band width attributable to variation in the preimpregnation parameters, (2) determination of the sensitivity of prepreg filament winding characteristics to variations of preimpregnating processing parameters, (3) definition of both the maximum and minimum degree of polymerization necessary for satisfactory winding, and (4) the effect of degree of polymerization on horizontal shear strength, NOL-ring tensile strength, strand strength, volatile content, etc.

A production lot of 20-end ECG 140/HTS roving was used for Phase II. This material is representative of the improved, high-strength glass roving in which the changes developed in Phase I were incorporated in a production run. The material was inspected for quality control on a sampling basis prior to

III Phase II - Preimpregnation Variable Study, A (cont.)

impregnation.

The resin system used for the Phase II program was the Shell 58/68R in place of E-787 resin employed for the development work in Phase I. This change was due to the proprietary nature of the E-787 composition. Since Phase II was basically a study of preimpregnation variables, it was necessary that the actual composition of the resin be known so that processing effects which are attributable to the resin system would be capable of interpretation. The Shell 58/68R resin system is formulated as follows:

| | |
|---|---|
| EPON 828 | 50 parts |
| EPON 1031-B80[*] | 61.5 parts |
| MNA (methyl nadic anhydride) | 90 PHR |
| BDMA (benzyldimethyl-amine) | Amount varied for the process variable studies |

A single lot of the above components was used to prepare batches of resin mix for the impregnation runs. Except for runs made on the same day with the same resin mix, fresh batches were prepared for all other runs just prior to impregnation.

1.  Process Variable Study

A total of 16 runs was made to investigate seven variables which affect the processing characteristics of prepreg. Five of these variables with the region of study are tabulated below:

| Process Variable | Region of Study |
|---|---|
| BDMA content, PHR | 0.35 to 0.75 |
| Running speed, ft/min | 55 to 83 |
| Tower temperature, $^\circ$F | 340 to 390 |
| Resin solution, sp. gr. at 75$^\circ$F | 0.833 to 0.863 |
| Resin bath temperature, $^\circ$F | 65 to 75 |

[*] 80% solution of EPON 1031 resin in MEK.

III Phase II - Preimpregnation Variable Study, A (cont.)

The study included investigation of at least three levels of each of the above process variables, except for the resin-bath temperature, in which two levels were investigated. Each parameter was investigated alone for its effect by maintaining all other parameters constant under a fixed processing condition. To a minor extent, interactions between the different parameters were investigated by changing two or more of the variables within the same run. The two remaining variables, creel tension and package geometry, were varied between rolls of roving within each run but held constant within a given roll.

For each run a set of parameters was predetermined and the conditions were held constant throughout the run. Variations in processing conditions were recorded and allowed to vary within their range of normal variation unless an unusually high variation occurred. When this happened, material prepared up to that point was discarded and the run restarted. Each run consisted of impregnating 10 rolls of roving for a total of 40 lb of prepreg. In addition, three control rolls were processed concurrently to check for resin pick-up, volatile content, and resin flow while in operation. The standard 3-1/8-in. waywind package was utilized for nine rolls of prepreg. The tenth roll was fabricated with a thread-wound pattern and a lead of 0.166 in. (60 turns per 10-in. core length). Five of the waywind rolls and the thread-wound roll were prepared at a tension of 2 lb applied at the creel. Of the other four rolls two waywind rolls each were prepared at a tension of 1.5 and 4 lb.

Changes during a run which occurred in the processing condition were followed by obtaining the gel time, specific gravity, and viscosity of the impregnating resin, and the temperature of the oven prior to, immediately after, and at 1/2-hr intervals during a run. At the start of the program two thermocouples were installed in each of the three towers. With a tower thermostat control and temperature recorder, three temperature readings were taken at predetermined time intervals as noted above. Thermocouples were located at 1/4 the distance from both ends of the tower so as to obtain a temperature profile of the tower.

III. Phase I - Preimpregnation Work to Mary, A (cont.)

2. Resin Content Study

Two types of devices were employed in an effort to thoroughly wet the roving and minimize variability of resin content in prepreg. One device, as shown in Figures 5 and 6, consisted of three rollers mounted on two end plates and was designed to apply pressure and to work the roving while immersed in the resin bath. The other device (Figures 7 and 8) was intended to pre-wet the roving and to permit resin penetration as it passed over a wet roller under a light pressure applied at the creel.

3. Band Width Control

This task was included to develop a procedure for measuring the band width of prepreg and to establish the dependence, if any, of band width upon the various parameters used in producing prepreg in the process variable study.

4. Processability on Aging

Rolls of prepreg from selected material lots were investigated for aging characteristics to establish the range of resin polymerization, as determined by viscosity index, necessary for satisfactory winding and its correlation with composite strength properties. A prepreg material was exposed to controlled atmospheric conditions, and at predetermined time intervals gel time and viscosity measurements were taken. Concurrently, composite test specimens which included NOL rings for both tensile and horizontal shear strength, were fabricated and the winding characteristics and physical changes of prepreg were observed.

5. Tests Applied to Prepreg

All incoming lots of prepreg material were inspected for quality control by the standard acceptance procedures. The tests included for all material lots were the determination of gravimetric data (volatile, resin content, and weight per linear yard of roving), strand tensile strength, and resin flow. Gel time and viscosity index tests were applied to determine the effects and sensitivity to variations in preimpregnating processing parameters. These two

III Phase II - Preimpregnation Variable Study, A (cont.)

tests were also applied to obtain aging data of prepregs exposed to a controlled atmospheric condition and for correlation with respect to prepreg winding characteristics. Procedures for the determination of viscosity index and gel time for prepregs are described in Appendixes A and B, respectively.

Band width measurements and resin content determinations within a roll were made in five locations spaced equally within a roll. A procedure developed for measuring the band width of prepreg roving is described in Appendix C.

B.    DISCUSSION OF TEST RESULTS

In order to evaluate prepreg material produced under various manufacturing parameters and to analyze the significance of the results obtained, a test method was necessary by which the processability of the prepreg could be measured quantitatively. The term processability as applied in this report is a generalized term which covers such diverse prepreg qualities as tack, greenness, fray, brittleness, etc., which are properties related to the resin. It is dependent upon three interacting properties, prepreg resin content, solvent content, and degree of polymerization. If these three properties are at a fixed level and nonvariant, the processability characteristics of the prepreg are nonvariant.

Standard methods exist for the measurement of resin and solvent content. However, a test method for degree of polymerization of prepreg resin was only developed recently.

The resin test method is a kinematic viscosity measurement of resin solution prepared by extraction of resin from prepreg with DMF (dimethylformamide). A detailed procedure established for this test is described in Appendix A. Essentially, the viscosity test is based on the premise that in solutions of the same concentration an increased viscosity is a function of increased degree of polymerization. Resins with higher degrees of polymerization offer a greater resistance to flow through a capillary tube in a viscometer and consequently result in longer efflux times. Figure 9 shows the results of a typical laboratory test that was performed in the development of this test method. In this test a

III Phase II - Preimpregnation Variable Study, B (cont.)

precise quantity of resin was apportioned in several equal parts and the resin advanced to different degrees of polymerization. Each resin sample was then dissolved completely in an accurately measured quantity of dimethylformamide (DMF). As shown in Figure 9, with known equal concentrations and varied degrees of resin advancement between the solutions, the refractive index remained constant, but the kinematic viscosity of each solution was measurably different. To demonstrate the sensitivity of refractive index to concentration, several known solutions of unequal concentrations were prepared and measurements taken. Figure 10 is a representation of the data obtained and indicates that the refractive index is a linear function of concentration. It was possible, therefore, to control the concentration of resin solution in DMF by means of a refractive index measurement. A viscosity measurement on the resin solution would then indicate relative degree of polymerization.

### 1. Processability and Aging of Prepreg

In addition to the evaluation of prepreg for the study of manufacturing parameters, processability of prepreg upon aging was investigated. Materials used in this study were those produced for the process variable investigation. These materials included a few lots of prepreg which were relatively green and had high tackiness. As prepreg was aged slowly at room temperature, viscosity and gel time measurements were taken and at the same time composite test specimens were fabricated. By means of this slow aging it was possible to follow the whole range cf prepreg processability characteristics and relate these to polymerization changes by means of the viscosity index.

The results of the effects of aging on viscosity, gel time, horizontal shear strength, and NOL-ring tensile strength are summarized in Tables 7, 8, 9, and 10 respectively. For a few selected material lots, correlations of the composite strengths with viscosity were made. Figures 11 to 17 show the changes in viscosity, horizontal shear strength, and NOL ring strength with aging time of the material exposed at room temperature. The figures indicate a definite relationship of composite strength properties with the viscosity index of prepreg. For both horizontal shear and NOL-ring tensile specimens, strength increases gradually with increase in viscosity until an optimum range of viscosity is attained.

III Phase II - Preimpregnation Variable Study, P (cont.)

Beyond this optimum range, strength drops off rapidly, generally at a rate faster than the original rate of strength increase. However, the rate of viscosity increase in this range is slow as compared with the earlier stage of advancement. Because of some fluctuations of test results, the optimum point of viscosity is not clearly defined, but from the number of tests conducted, a tentative range of viscosity for optimum strength properties may be determined - e.g., the data indicate that a viscosity for optimum NOL-ring tensile strength and for the horizontal shear strength is obtained at a range of 1.85 to 1.95 viscosity units and 1.90 to 2.00 viscosity units, respectively. At this viscosity range prepreg is still within a region of good processing characteristics but is approaching a region of low tack.

It should be noted that these relationships hold only for the particular process used to prepare the composites. If a different process were used such as increased addition of heat during winding, the curves would be displaced.

Some of the variation that has been obtained in horizontal shear strength of incoming material for production can be explained by the fact that composite strength is dependent upon the viscosity of prepreg system at the time of fabrication. Qualitative observation of incoming material indicates that the spread in the degree of polymerization of standard prepreg is large enough to affect horizontal shear strength.

No good correlation of the gel time data to processability or to degree of resin advancement in prepreg could be made. Generally, the results were erratic and the changes observed with aging time were small. Further discussion of the gel-time data is presented in the following section of this report.

The effect of aging on strand strength and volatile content of prepreg was also investigated. Past experiences have indicated that, besides the basic glass strength, AeroROVE prepreg strand strength was dependent upon the state of resin advancement at the time of specimen preparation. The results obtained from the first few lots of material, shown in Tables 11 and 12, did not indicate any significant changes with time, and testing was discontinued for the

III Phase II - Preimpregnation Variable Study, B (cont.)

subsequent aging studies.

Viscosity data in relation to aging for a few selected runs is presented in Figure 18. It can be seen that the shape of all curves for materials containing the same amount of BDMA is very similar. This indicates that for a given BDMA content, the rate of resin advancement or polymerization is relatively constant at a constant temperature. Therefore, any storage life for the material which is used up during B-staging in the oven cannot be recovered.

Figure 19 is a composite curve made from a series of actual aging curves of the type shown in Figure 18. Figure 18 indicates the spectrum of processability changes which prepreg undergoes at room temperature, assuming initially that the prepregs were green or insufficiently B-staged. It should be noted that the optimum range of viscosity for filament winding, as shown in Figure 18, to be between 1.82 and 1.96 centistokes, is tentative since differences in resin content and other associated qualities of the prepreg will alter the handling and winding characteristics.

It should be noted also that the optimum range of viscosity index shown in Figure 19 assumes a constant fabrication process. The fabrication process could be modified such that materials of high viscosity index could be usable. As an example, the material could be modified by application of heat or by other means to reduce the viscosity to make the material applicable for filament winding. However, such a modification of the fabrication process to suit material requirements is undesirable under production conditions from the standpoint of reproducibility, reliability, and performance.

Figure 19 also suggests a possible approach to a reproducible and predictable B-staged material for any degree of resin advancement desired to suit any particular process as well as a prepreg with maximum storage life. This approach would be to produce a material which is slightly under-B-staged and to allow it to advance slowly under controlled conditions until the desired processability properties are obtained. The viscosity index could be used to follow these changes and define the point at which the material has optimum winding properties.

III Phase II - Preimpregnation Variable Study, B (cont.)

2. Process Variable Study

A summary of laboratory test data for all incoming material
produced under various processing parameters for this study is presented in
Table 13. These same lots of material were subjected to outgoing quality-control
inspection at the manufacturer's facility. This test data with the measurements
of the process conditions taken during the manufacture of these materials is
summarized in Table 14. To simplify and facilitate the analysis of the test
results, the data shown in Table 13 has been reduced and combined into appropriate
groups to illustrate the effect and sensitivity of various process parameters
upon qualities of prepreg. Tables 15, 16, and 17 show the effect of resin con-
tent, viscosity, and gel time of a prepreg, respectively. Also, corresponding
data is graphically illustrated in Figures 20 through 30.

a. Specific Gravity

Figure 20 shows the effect of specific gravity of the
impregnating resin bath on resin content of prepreg. In the region of specific
gravity, investigated with all other variables held constant at a nominal level,
resin content of prepreg is increased by 0.22% for an increase of 0.001 units of
specific gravity as measured with a hydrometer at 75°F. This direct relationship
is expected, since the concentration of a solution is a function of specific
gravity and greater amounts of resin solids are deposited on a roving for a given
volume of solution. Also, with increased concentration, the viscosity of a
solution correspondingly increases. This increase will have a direct effect on
the amount of resin picked up on a roving.

b. Running Speed

As shown in Figure 21, resin content is also related
directly to the speed with which a roving is passed through the impregnation sys-
tem. The increase in resin content is related to the reduction in drainage time
of excess solution as compared to slower speeds. The change in resin content was
approximately 0.13% increase for an increase of 1 ft per minute of running speed.

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####    c.      Flow

Within a range of viscosity satisfactory for filament
winding, resin flow is related directly to resin content of prepreg. Figure 22
shows a 1.1% change in resin flow for a change of 1% resin content. The slight
deviation of flow data from the curve may be traced to a large difference in
viscosity index for the material lots. Slightly higher resin flow is obtained
from prepregs which are less advanced (B-staged) than with prepregs of equi-
valent resin content with a higher degree of advancement. However within the
usual range of resin content and degree of polymerization in which prepreg is
used, resin content is the major controlling influence on flow.

####    d.      Viscosity

As discussed previously, the viscosity test has been
used to measure the relative degree of resin polymerization and has been satis-
factorily correlated to the processability of prepreg. The viscosity test was
also applied to determine the effect of various amounts of BDMA, running speed,
and tower temperature on the B-staging of the prepreg resin. In Figure 23 the
effect of tower temperature on the viscosity index of the prepreg is shown. These
lots of prepreg were all produced under a constant running speed with the resin
containing the same amount of BDMA accelerator. It can be seen that the vis-
cosity index is directly related to the amount of heat in-put during the manu-
facturing process as measured by the tower temperature. Figure 23 shows that
for an increase of $10^{\circ}F$ tower temperature, viscosity is raised by 0.044 unit.
This is equivalent to one day of aging at room temperature (Figure 18). As noted
in Table 14, a considerable temperature spread was obtained within a tower when
the temperature was measured at three locations and between the towers. Since
the deviation of temperature from the fixed condition was significant, actual
measured temperatures were used instead of the fixed condition for plotting the
data. An average temperature of the actual readings taken from both the top
thermocouple and the control gage of the first two towers was used. Although the
temperature of the third tower varied between runs from a nominal condition of
$150^{\circ}F$, adjustments were not made for these differences in the calculation of
average temperature. Small temperature differences at this low level have a

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relatively minor effect on viscosity as compared with the temperature effect
at elevated temperatures.

Figure 24 shows the effect of running speed on viscosity
of prepreg produced under a constant tower temperature. Again, the viscosity
change is shown to be related directly to the heat in-put during the manufacturing
process. With increased running speed, dwell time in the treater towers is re-
duced and the resultant effect is indicated by a lower viscosity index:  the
viscosity change was 0.009 centistoke decrease for an increase of 1 ft per min-
ute of running speed. For this data analysis, viscosity was adjusted to correct
for the temperature variations obtained between runs. The relationship obtained
in Figure 23 was applied to adjust the data to a nominal temperature of $365^{\circ}$F.
The relationship of running speed to tower temperature is such that a variation
of 1 ft per minute running speed is equivalent to a $2^{\circ}$F change in tower tem-
perature at a nominal temperature of $365^{\circ}$F. It should be noted, however, that
all the data may be applicable only to the particular set of equipment and pro-
cessing conditions utilized for this study. However, this approach to the develop-
ment of data for process control has a general applicability.

e.   Gel Time

As with the analysis of viscosity data, the effect of
tower temperature and running speed on gel time is shown in Figures 25 and 26,
respectively. A nonlinear relationship appears to exist for the effect of tower
temperature on gel time. At a higher processing temperature the resin is advanced
to a higher level of B-stage, and the gel time is correspondingly lowered. How-
ever, the rate of change in gel time is reduced considerably as the temperature
is increased. The data suggests that the polymerization reaction is highly sen-
sitive to small changes in temperature. The reaction progresses rapidly in the
early stages to a level in which resin changes become less sensitive to tempera-
ture effects. One possible explanation for this phenomenon could be that the
resin at a higher level of B-stage becomes more viscous, and the reaction may be
subject to diffusion control rather than kinetic or thermodynamic control. Another
possible explanation is that some fraction of the original amount of BDMA is
volatilized during the heat treatment. Comparison of the gel-time data for

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virgin resin mixes (Table 14) and prepregs (Table 16) provides substantial evidence of this behavior. In some cases, longer gel time is obtained from prepreg than for the same resin mix from which the prepreg was made. Also, data to support the contention of BDMA volitilization is shown in Figures 27 and 28. Figure 27 shows that gel time is definitely affected by the amount of BDMA contained in the original resin mix and, even upon aging, the gel time is distinctly different (dependent on BDMA content). Figure 28, correlating gel time with aging, indicates a similar type of dependence of gel time as obtained in Figure 27, but the magnitude of difference was smaller. These prepregs were produced with a resin mix containing the same amount of BDMA but processed under various processing conditions.

The effect of running speed on gel time is shown in Figure 26. As with the viscosity data, adjustments were made to gel-time data to correct for the variation in tower temperature between runs. Based on Figure 25, a correction factor of 0.3 sec per 1°F difference in tower temperature was applied. Even with this adjustment, a large scatter in gel time was obtained. These scatters were especially evident in the aging study.

f.    Effect of BDMA

Three levels of BDMA accelerator content in the basic resin formulation were initially investigated to establish the optimum content for prepreg application. Strength properties of the composites and reactivity of prepreg for both aging and storageability were considered for the selection of BDMA content to be used for the remainder of the runs. Data on gel time for various amounts of BDMA is presented in Table 11, and corresponding data is shown graphically in Figure 29. It can be seen that the reactivity of the Shell 58/68R resin system is more sensitive to small changes in the amounts of BDMA at a lower range of BDMA content. Composite strength in horizontal shear is slightly superior with higher BDMA content, but not significantly different in the NOL-ring tensile strength.

g.    Correlation of Gel Time and Viscosity

The data indicates that the gel-time test is not so

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reproducible or reliable as the viscosity test, and no direct correlation can
be made of the gel-time data to the processability of prepreg. However, the
value of a gel-time test lies in the determination of the reactivity of a pre-
preg system and, as such, compliments the viscosity data. Figure 30, showing
the relationship of gel time and viscosity, was derived from data presented
previously in Figures 23 through 26. The large scatter of points shown in Figure
30 is due in part to the large variance in gel-time data and in part to the
deviations of viscosity data. In spite of the scatter, however, the type of
curve shown in Figure 30, is useful for material inspection and can be applied
for process optimization of prepregs. An example of the type of material in-
spection which can be applied is shown in Figure 30 for a prepreg containing
0.75 PHR BDMA. In this case it is noted that the gel time is considerably lower
than is usual for the corresponding viscosity. This would immediately show that
the material is too reactive and that storageability or bench life of the material
would be appreciably shortened.

3.    Resin Content Study

In order to reduce the variability of resin content in pre-
pregs, special devices were developed for this program and applied as described
previously in the section on test procedure. Laboratory test data of the two
runs produced by utilization of these devices is summarized in Table 18. Based
on standard deviation of 0.48 and 0.34% as compared with 0.43% of the standard-
type material lots (Table 15), the variability of resin content between rolls
is not improved significantly from other material lots produced without the
special devices.

Table 19 summarizes the data of resin content in prepreg for
various tensions applied at the creel. The data shows a slight trend to a higher
resin content with 1.5-lb creel tension as compared with 2.0 and 4.0-lb creel
tension, especially for process conditions used to produce high resin content
(above 23%) prepregs. Generally, however, the results were inconsistent. In
order to gain a better understanding of the effect of tension on resin content
and the inconsistencies obtained, roving tensions were measured at various loca-
tions between contact points while in operation. These measurements, summarized

III Phase II - Preimpregnation Variable Study, B (cont.)

in Table 20, indicate that tension introduced at the creel is immediately altered
after the first contact point (as shown in Figure 2 for the schematic diagram
of the preimpregnation line) and that tension varies with time within the same
location.  If roving tension is one of the contributing factors which affect the
resin content in prepregs, variations in roving tension as shown in Table 20 may
be a partial explanation for the variations observed within a material lot and
even within a roll.

4.　Package Geometry

For each impregnation run one of the 10 prepreg rolls was
wound with a thread-wound pattern with a lead of 0.116 in. as compared with a
3.125-in. lead of the standard waywind package.  The objective of this study was
to determine whether any improvement or benefit is gained from a reduced packaging
lead angle over the standard pattern.  Comparative results of the AeroROVE strand
test and fiber tensile strength calculated from the NOL-ring composite tensile
strength are presented in Table 19.  Strengthwise, there is no significant dif-
ference between the two packages, but uniformity in band width within a roll is
superior with a thread-wound package.  No sudden band width changes, such as
those which occur at the traverse termination points of a waywind package, were
observed, and no unusual winding problem was experienced during the specimen
fabrication.  However, the package had a tendency to flow out and deform the pack-
age shape under a slight take-up winding tension, more so than for a standard
waywind package.  This condition was more clearly evident when winding a "green"
prepreg.  It was not possible to evaluate the influence of this factor (package
deformation) on processability and prepreg quality during this program.

5.　Band Width

A relatively simple procedure has been developed, as described
in Appendix C, for the measurement of the band width of prepreg roving.  Three
measurements of band width were taken for each specimen as follows:  (1) width
as unspooled directly from a package to represent the dependence upon the roving
and the processing parameters used to produce prepreg, (2) width under tension
to represent a fabrication condition where winding tension is normally applied,

III Phase II - Preimpregnation Variable Study, B (cont.)

and (3) width at the table edge under tension to represent a condition immediately after being applied on the part. Band width measurements between rolls and within single rolls of prepreg are shown in Table 21. A definite change in band width is noted for the three conditions of measurement. The amount of change appears to be related directly to resin content and inversely to the viscosity index of the prepreg. Since prepreg processing variables affect both resin content and the degree of polymerization, it may be concluded that band width roving is dependent upon the various parameters used in producing prepreg.

C. CONCLUSIONS

The processing characteristics of prepreg have been shown to be very sensitive to levels of manufacturing parameters used in producing prepregs. Test data shows that resin content is affected directly by process variables such as specific gravity of the impregnating resin solution, running speed, and, to some extent, tension applied at the creel. Also, bandwidth is affected by variations in both resin content and degree of polymerization, which in turn is dependent on the processing parameters. The most significant single property of prepreg which affects processability is the degree of resin polymerization (B-staging) if the resin and solvent content are relatively constant. Manufacturing parameters which affect this property are tower temperature and running speed, both of which are related to the amount of heat-energy input to the material during the manufacture. However, in spite of these relationships and their sensitive dependence on levels of process variables, very uniform and predictable prepregs can be made if manufacturing parameters are closely controlled and maintained.

An approach to setting quantitative limits for prepreg manufacturing has been demonstrated by the application of the viscosity-index test as a means of assigning differences in processing characteristics of prepregs. This study has also demonstrated the critical need for measuring degree of polymerization during the early stages in the application of any new resin to prepreg. Although levels of manufacturing parameters defined in this program are for a specific process, this approach has general applicability to other prepreg manufacturing processes.

III Phase II - Preimpregnation Variable Study, C (cont.)

This is the first time that quantitative data of this type defining the manufacture of materials, has been developed, and it is considered a significant breakthrough in the development of scientifically controlled processes and materials in the field of reinforced plastics. Further studies recommended in this area of development would be investigations of interactions between prepreg processing variables and relationships of prepreg processability to the strength of filament-wound internal pressure vessels.

TABLE 1

Table 1 a

**CHARACTERISTICS AND STRENGTH DATA OF DRY EXPERIMENTAL ROVING**

Table 1 b

Table 1 c

## TABLE 1 (cont.)

Table 1 d

GLAZING AND STRENGTH DATA OF DRY EXPERIMENTAL ROVING

Table 1 e

Table 1 f

## TABLE 1 (cont.)

Table 1 g

GRAVIMETRIC AND STRENGTH DATA OF DRY EXPERIMENTAL ROVING

Table 1 h

TABLE 2

Table 2 a

CHARACTERISTICS AND STRENGTH DATA OF EXPERIMENTAL PREFORMING ROVING

Table 2 b

## TABLE 2 (cont.)

Table 2 c

CHARACTERISTIC AND STRENGTH DATA OF EXPERIMENTAL PREFIRED ROVING

Table 2 d

## TABLE 2 (cont.)

Table 2 e

**GRAIN WEIGHT AND STRENGTH DATA OF EXPERIMENTAL PREPREG ROVING**

Table 2 f

| Lot No. 5 | Ultimate Tensile Strength (Strand) (ksi) | | | Loss on Ignition (Resin Solids) (%) | | | Volatile Content (%) | | | NOL Ring (Glass Stress), (ksi) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Roll No. | Av | High | Low | Av | High | Low | Av | High | Low | Av | High | Low |
| 3 | 308.1 | 313.4 | 293.8 | 19.73 | 19.92 | 19.42 | 1.97 | 2.18 | 1.81 | 342.4 | 350.5 | 332.7 |
| 4 | 299.3 | 328.9 | 283.9 | 20.40 | 21.36 | 19.90 | 1.92 | 1.94 | 1.90 | 340.2 | 355.0 | 328.5 |
| 5 | 300.7 | 315.0 | 265.4 | 19.00 | 19.32 | 18.64 | 1.83 | 1.86 | 1.82 | 340.8 | 344.5 | 334.6 |
| 7 | 342.5 | 365.8 | 295.4 | 18.58 | 18.97 | 17.98 | 1.88 | 2.07 | 1.71 | 342.7 | 351.9 | 333.4 |
| 8 | 316.0 | 337.0 | 295.0 | 19.30 | 19.56 | 19.05 | 1.66 | 1.82 | 1.46 | 361.0 | 368.9 | 352.8 |
| 9 | 313.5 | 325.8 | 303.1 | 19.09 | 19.41 | 18.63 | 1.85 | 1.96 | 1.83 | 346.8 | 355.0 | 352.8 |
| 10 | 316.9 | 334.4 | 282.4 | 19.18 | 19.68 | 18.75 | 1.99 | 2.08 | 1.95 | 350.9 | 356.1 | 334.1 |
| 11 | 340.7 | 361.1 | 312.5 | 19.42 | 19.80 | 19.38 | 1.91 | 2.19 | 1.70 | 352.5 | 358.1 | 347.4 |
| 12 | 345.4 | 353.0 | 340.3 | 19.88 | 20.48 | 18.51 | 1.62 | 1.94 | 1.10 | 355.4 | 365.8 | 345.2 |
| 13 | 354.1 | 365.3 | 305.8 | 19.64 | 18.87 | 18.97 | 1.99 | 2.07 | 1.94 | 355.6 | 370.0 | 339.7 |
| 14 | 329.1 | 356.1 | 290.8 | 18.69 | 18.87 | 18.56 | 2.18 | 2.29 | 2.06 | 358.1 | 363.8 | 348.2 |
|  |  |  |  |  |  |  |  |  |  |  |  | 352.7 |
| Lot Average | 323.8 | | | 19.35 | | | 1.39 | | | 349.7 | | |
| Av Deviation | 15.0 | | | 0.41 | | | 0.11 | | | 4.4 | | |

| Lot No. 6 | Ultimate Tensile Strength (Strand) (ksi) | | | Loss on Ignition (Resin Solids) (%) | | | Volatile Content (%) | | | NOL Ring (Glass Stress), (ksi) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Roll No. | Av | High | Low | Av | High | Low | Av | High | Low | Av | High | Low |
| 9 | 304.8 | 315.2 | 284.5 | 24.19 | 24.30 | 24.10 | 2.53 | 2.58 | 2.47 | 315.6 | 330.0 | 305.0 |
| 10 | 297.7 | 312.9 | 286.4 | 25.16 | 25.44 | 24.92 | 2.27 | 2.35 | 2.11 | 329.5 | 342.0 | 316.5 |
| 11 | 305.1 | 318.5 | 295.4 | 24.31 | 24.58 | 24.18 | 2.46 | 2.59 | 2.31 | 326.3 | 337.5 | 314.4 |
| 12 | 321.9 | 352.4 | 288.9 | 21.10 | 21.37 | 20.84 | 2.02 | 2.07 | 1.96 | 316.3 | 331.5 | 297.9 |
| 13 | 310.7 | 337.6 | 292.1 | 22.60 | 23.10 | 21.88 | 2.16 | 2.25 | 2.03 | 310.0 | 315.9 | 299.3 |
| 14 | 314.2 | 326.9 | 298.8 | 21.81 | 21.93 | 21.68 | 2.16 | 2.28 | 2.11 | 312.4 | 317.4 | 309.8 |
| 15 | 319.1 | 327.2 | 306.5 | 23.92 | 23.24 | 23.58 | 2.34 | 2.43 | 2.28 | 313.3 | 324.0 | 298.4 |
| 16 | 305.5 | 343.3 | 283.4 | 22.88 | 23.22 | 22.57 | 2.45 | 2.48 | 2.42 | 304.9 | 307.9 | 300.9 |
| 17 | 330.5 | 345.5 | 314.5 | 24.16 | 24.30 | 23.89 | 2.27 | 2.44 | 2.15 | 310.1 | 324.0 | 296.4 |
| 18 | 322.2 | 390.3 | 314.2 | 23.48 | 23.67 | 23.37 | 2.09 | 2.16 | 1.99 | 306.1 | 311.0 | 299.3 |
| 19 | 317.1 | 334.4 | 284.7 | 22.71 | 22.86 | 22.47 | 1.92 | 2.04 | 1.80 | 308.0 | 313.9 | 304.8 |
| 20 | 311.6 | 363.5 | 265.4 | 21.34 | 21.61 | 20.56 | 1.86 | 1.95 | 1.77 | 309.2 | 318.9 | 300.6 |
| 21 | 339.4 | 348.4 | 318.8 | 21.51 | 21.73 | 21.03 | 1.80 | 2.00 | 1.59 | 305.7 | 320.5 | 300.0 |
| Lot Average | 315.4 | | | 23.03 | | | 2.18 | | | 313.0 | | |
| Av Deviation | 8.8 | | | 1.08 | | | 0.19 | | | 5.6 | | |

## TABLE 2 (cont.)

Table 2 g

COMPOSITION AND STRENGTH DATA OF EXPERIMENTAL PREPREG ROVING

Table 2 h

TABLE 3

18-in. DIAMETER FILAMENT WOUND CHAMBERS - TEST RESULTS

## TABLE 4

### EFFECT OF VINYL COATING ON PREPREG STRAND STRENGTH

| | | Ultimate Tensile Strength (psi x $10^{-3}$) | | | | | |
| | | Standard Prepreg Strand | | | Vinyl-Coated Prepreg Strand | | |
| Lot No. | Roll No. | Average | High | Low | Average | High | Low |
|---|---|---|---|---|---|---|---|
| 3-804 | 19 | 293.4 | 310.2 | 272.4 | 377.2 | 391.2 | 352.0 |
| 4-826 | 7 | 324.8 | 358.9 | 310.6 | 393.1 | 406.3 | 371.5 |
| 5-838 | 12 | 345.4 | 353.0 | 340.3 | 381.2 | 408.6 | 338.0 |
| 7-846 | 7 | 353.1 | 365.8 | 335.8 | 399.7 | 412.0 | 377.3 |

## TABLE 5

### OWENS-CORNING QUALITY CONTROL DATA FOR EXPERIMENTAL GLASS

| Lot No. | Stiffness | Volatile Content (%) | Solids Content (%) | Wet-Out Rate | Package Hardness | Fuzz Content (x10) | Resin Content (%) Avg. | High | Low |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 3.88±0.11 | 0.204±0.010 | 1.12±0.03 | 89.50±1.50 | 66.1±3.5 | 0.08±0.045 | 20.51±0.84 | 21.82 | 19.47 |
| 2 | 3.20±0.11 | 0.015±0.004 | 1.21±0.07 | 89.70±1.48 | 68.6±1.7 | 1.54±0.65 | 18.98±0.85 | 20.80 | 17.20 |
| 3 | 3.57±0.07 | 0.106±0.015 | 1.19±0.03 | 86.76±2.16 | 69.2±1.7 | 1.64±0.60 | 19.83±0.95 | 22.17 | 18.50 |
| 4 | 3.26±0.19 | 0.246±0.017 | 1.06±0.03 | 85.89±1.33 | 74.5±1.7 | 2.75±0.50 | 18.21±1.28 | 19.92 | 15.01 |
| 5 | 3.49±0.11 | 0.106±0.035 | 1.23±0.04 | 89.07±1.67 | 69.3±1.6 | 1.88±0.38 | 19.39±0.33 | 20.40 | 18.58 |
| 6 | 3.80±0.10 | 0.021±.007 | 1.30±0.08 | 97.75±1.20 | 71.2±2.5 | 0.0033±0.0006 | 23.03±1.08 | 25.16 | 21.10 |
| 7 | 2.98±0.08 | 0.165±0.018 | 1.30±0.04 | 92.38±1.88 | 74.3±2.8 | 4.60±0.50 | 20.10±0.26 | 20.58 | 19.67 |
| 8 | 3.82±0.10 | 0.35±0.006 | 1.31±0.05 | 97.00±0.30 | 76.2±4.0 | 0.0038±0.0013 | 22.13±1.25 | 23.25 | 19.12 |

## TABLE 6

### ULTIMATE TENSILE STRENGTH OF PRODUCTION PREPREG

| Date | Lot. No. | No. Rolls Tested | Strand Tensile Strength (psi) | | |
| --- | --- | --- | --- | --- | --- |
| | | | Average | High | Low |
| April 1962 | 89/F911 | 13 | 337 | 359 | 298 |
| April 1962 | 90/F911 | 13 | 342 | 353 | 319 |
| May 1962 | 91/F917 | 15 | 338 | 349 | 306 |
| May 1962 | 92/F923 | 26 | 346 | 372 | 310 |
| May 1962 | 94/F935 | 13 | 362 | 400 | 329 |
| June 1962 | 95/F942 | 15 | 400 | 419 | 370 |
| June 1962 | 96/F944 | 15 | 396 | 405 | 376 |
| June 1962 | 97/F950 | 15 | 373 | 388 | 335 |
| July 1962 | 100/F961 | 15 | 383 | 406 | 328 |
| July 1962 | 101/F965 | 15 | 382 | 405 | 353 |
| July 1962 | 103/F973 | 15 | 378 | 402 | 352 |
| July 1962 | 105/F971 | 15 | 374 | 399 | 353 |
| July 1962 | 107/F979 | 14 | 376 | 414 | 360 |
| August 1962 | 106/F976 | 15 | 381 | 396 | 352 |
| August 1962 | 108/F983 | 15 | 367 | 390 | 339 |

Table 6

## TABLE 7

Table 7

## TABLE 8

### GEL TIME WITH AGING AT ROOM TEMPERATURE

| Run No. | 1 | 2 | 3 | 8 | 9 | 10 | 13 | 14 | 15 | 16 |
|---|---|---|---|---|---|---|---|---|---|---|
| Lot No. | F-1070 | F-1070 | F-1070 | F-1102 | F-1102 | P-1118 | P-1118 | P-1154 | P-1154 | P-1155 |
| Process Variable | .35 EDMA | Nominal | .75 EDMA | 55 ft/min | 75 ft/min | 83 ft/min | 340°F | 80 ft/min | 345°F | 145 EDMA; 350°F |

GEL TIME, SEC

| Aging Time, Days | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 0 | 193 | 142 | 108 | 106 | 119 | 177 | 167 | 176 | 166 | 170 |
| 1 | 202 | 151 | 118 | 101 | 131 | . | . | 164 | 152 | 184 |
| 2 | 183 | 134 | 119 | 101 | 117 | . | . | 170 | 154 | 160 |
| 3 | 179 | 150 | 90 | . | . | . | . | 182 | 169 | 169 |
| 4 | 157 | 138 | 96 | . | . | 119 | 109 | 155 | 155 | 149 |
| 5 | 155 | 134 | 88 | 107 | 127 | 119 | 107 | 165 | 134 | 153 |
| 6 | 162 | 138 | 96 | 101 | 130 | 120 | 101 | 164 | 145 | 147 |
| 7 | . | 137 | 102 | 109 | 118 | 118 | 97 | 161 | 155 | 165 |
| 8 | 155 | 130 | 74 | 103 | 125 | . | . | 148 | 148 | 152 |
| 9 | . | . | . | 97 | 118 | . | . | 151 | 148 | 145 |
| 10 | . | . | . | . | . | 96 | 92 | 150 | 136 | 155 |

Table 8

## TABLE 9

**HORIZONTAL SHEAR STRENGTH WITH AGING AT ROOM TEMPERATURE**

| Run No. | 1 | 2 | 3 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lot No. | P-1070 | P-1070 | P-1070 | P-1102 | P-1102 | P-1118 | P-1108 | P-1108 | P-1118 | P-1154 | P-1154 | P-1155 |
| Process Variable | 0.35 PSM | Nominal (.55) | 0.75 PSM | 55 ft/min | 75 ft/min | 65 ft/min | 390°F | ?00? 75 ft min | 340°F | 80 ft/min | 345°F | .45 PSM 350°F |
| **Aging Time, Days** | | | | | | | | HORIZONTAL SHEAR STRENGTH, psi (1) | | | | |
| 0 | 9770 | 11150 | 10640 | 11420 | 9420 | 9920 | 9440 | 11610 | 10000 | 8000 | 7820 | 9020 |
| 1 | 9210 | 10120 | 11050 | - | 11910 | 10820 | - | 11450 | 9550 | 8470 | 8450 | 9330 |
| 2 | 11320 | 10930 | 11580 | 11750 | 10860 | - | 11110 | 12040 | - | - | - | - |
| 3 | 9140 | 11550 | 11360 | 12100 | 12090 | 12410 | 11010 | - | 11860 | 9830 | 10260 | 8980 |
| 4 | 10840 | 11540 | 11570 | - | - | 11250 | 11280 | 12460 | 12120 | 9420 | 10640 | 9270 |
| 5 | 10080 | 11640 | 11800 | 11820 | 12130 | 12190 | - | 12620 | 12760 | 9670 | 9560 | 9970 |
| 6 | 10710 | 11010 | 11230 | 10610 | 12100 | 11590 | - | 12730 | 11790 | 10280 | 10920 | 10300 |
| 7 | 10150 | 11650 | 11870 | 11590 | 12030 | 11650 | 10360 | 10800 | 11580 | 10830 | 10650 | 11280 |
| 8 | 11250 | 11570 | 11850 | 11000 | 11450 | 11680 | 10490 | 10620 | 11580 | 10880 | 11230 | 10170 |
| 9 | 10880 | 11340 | 11340 | 10530 | 11310 | - | 9800 | 10850 | - | - | - | - |
| 10 | 11440 | 11280 | 11220 | 10130 | 10870 | 11550 | 6530 | - | 10850 | 10300 | 9590 | 8520 |
| 11 | | | | | | | | | | 8270 | 4470 | 5240 |
| 12 | | | | | | | | | | 7990 | 2980 | 3930 |

(1)  Data represents an average of three (3) test specimens. All specimens were tested at room temperature after an initial 6 hour aging cycle. The loading rate was based on a head-travel speed of 0.5 in/minute. For Run Nos. 1, 2 and 3 material was heated to 150°F prior to and during winding of prepreg. No heat was applied to the remainder of the runs.

Table 9

## TABLE 10

### NOL-RING COMPOSITE AND FIBER TENSILE STRESSES
### WITH AGING AT ROOM TEMPERATURE

NOL Min.(1) Tensile Strength, $10^3$ psi

| Run No. | 1 | | 2 | | 3 | | 8 | | 9 | | 10 | | 13 | | 14 | | 15 | | 16 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lot No. | P-1070 | | P-1070 | | P-1070 | | F-1102 | | F-1102 | | F-1118 | | F-1118 | | F-1154 | | F-1154 | | F-1155 | |
| Process Variable | .35 RPM | | (Nominal) | | .75 RPM | | 55 ft/min | | 75 ft/min | | 83 ft/min | | 340°F | | 65 ft/min | | 345°F | | .045 RPM | |
| Aging Time, Days | Comp. | Fiber | Comp. | Fiber | Comp. | Fiber | Comp. | Fiber | Comp. | Fiber | Comp. | iber | Comp. | Fiber | Comp. | iber | Comp. | Fiber | Comp. | Fiber |
| 0 | 220.7 | 356.0 | 219.6 | 354.4 | 218.0 | 352.8 | 216.0 | 341.5 | 214.1 | 365.2 | 192. | 347.8 | 203.0 | 352.0 | 211.0 | 351.0 | 225.9 | 352.3 | 224.6 | 347.9 |
| 1 | 211.8 | 335.7 | 218.1 | 339.0 | 217.2 | 344.5 | 216.0 | 344.0 | 209.5 | 344.1 | 195.0 | 344.0 | 270.3 | 327.9 | 220.7 | 345.0 | 235.6 | 344.4 | 232.0 | 351.0 |
| 2 | 222.8 | 348.3 | 215.6 | 345.6 | 220.1 | 351.0 | 224.2 | 351.0 | 222.0 | 335.0 | – | – | – | – | – | – | – | – | – | – |
| 3 | 215.9 | 346.8 | 214.0 | 344.0 | 213.7 | 346.0 | 221.4 | 340.2 | 223.2 | 350.9 | 199.0 | 347.0 | 203.7 | 341.1 | 215.0 | 346.1 | 224.3 | 351.3 | 224.3 | 351.3 |
| 4 | 215.9 | 345.0 | 204.7 | 338.2 | 214.9 | 343.9 | – | – | – | – | 212.0 | 352.5 | 206.3 | 354.0 | 247.0 | 345.0 | 225.0 | 345.0 | 227.8 | 345.4 |
| 5 | 222.5 | 353.5 | 203.6 | 329.3 | 214.8 | 352.0 | 208.4 | 332.9 | 222.7 | 343.2 | 186.0 | 342.5 | 206.5 | 352.2 | 217.0 | 331.7 | 219.9 | 339.2 | 223.9 | 354.4 |
| 6 | 211.0 | 345.0 | 212.5 | 347.0 | 194.7 | 329.9 | 210.6 | 329.0 | 216.1 | 346.9 | 180.0 | 349.9 | 217.2 | 344.0 | 233.0 | 345.2 | 225.9 | 341.1 | 223.9 | 340.0 |
| 7 | 211.5 | 344.3 | 211.7 | 351.6 | 195.9 | 349.3 | 224.6 | 347.0 | 229.0 | 359.6 | 186.3 | 343.3 | 215.4 | 343.1 | 222.5 | 355.5 | 225.7 | 341.8 | 225.2 | 335.0 |
| 8 | 214.7 | 350.1 | 215.8 | 352.1 | 211.2 | 341.0 | 201.1 | 341.1 | 205.8 | 340.3 | 169.0 | 340.7 | 219.4 | 341.1 | 227.0 | 341.1 | 221.1 | 345.0 | 213.1 | 341.8 |
| 9 | 207.7 | 335.8 | 204.4 | 334.0 | 203.4 | 333.0 | 174.0 | 337.9 | 198.4 | 330.0 | – | – | 215.0 | 340.1 | – | – | – | – | – | – |
| 10 | 219.5 | 353.4 | 204.1 | 323.5 | 299.2 | 335.6 | 247.0 | 329.2 | 204.9 | 352.6 | 190.3 | 335.3 | 1.03 | 347. | 1.03 | 340.1 | 211.0 | 340.5 | 192.0 | 320.5 |
| 11 | | | | | | | | | | | | | 192.6 | 324.1 | 192.6 | 324.1 | 172.2 | 340.1 | 132.7 | 319.2 |
| 12 | | | | | | | | | | | | | 1.30 | 2. | 1.30 | 2. | 1.30 | 340.2 | 150.0 | 290.0 |

(1) NOL ring specimens were prepared with 23 turns of roving wound with 1½ pound tension for a nominal composite thickness of 0.600 inch. For run nos. 1,2, and 3 the mandrel was heated to 150°F prior to and during winding. No heat was applied to the remainder of the runs. Data represents an average of three (3) test specimens.

Table 10

TABLE 11

AEROROVE STRAND STRESS WITH AGING AT ROOM TEMPERATURE

| Run No. | 1 | 2 | 3 | 8 | 9 |
|---|---|---|---|---|---|
| Lot No. | F-1070 | F-1070 | F-1070 | F-1102 | F-1102 |
| Process Variable | .35 BDMA | Nominal | .75 BDMA | 55 ft/min | 75 ft/min |

AEROROVE STRAND TENSILE STRENGTH, $10^3$ psi

| Aging Time, Days | | | | | |
|---|---|---|---|---|---|
| 0 | 327.2 | 350.8 | 361.4 | 345.8 | 354.2 |
| 1 | 312.9 | 372.7 | 352.0 | 318.1 | 273.4 |
| 2 | 327.2 | 353.9 | 344.9 | 325.2 | 332.8 |
| 3 | 356.5 | 382.6 | 358.4 | - | - |
| 4 | 341.5 | 359.7 | 351.3 | - | - |
| 5 | 348.2 | 337.2 | 388.0 | 296.7 | 312.6 |
| 6 | 350.6 | 385.6 | 349.7 | 339.4 | 360.1 |
| 7 | 323.6 | 398.5 | 370.9 | - | - |
| 8 | 361.3 | 368.5 | 371.5 | 339.4 | 378.4 |
| 9 | 321.2 | 357.2 | - | - | - |
| 10 | 329.6 | 354.4 | - | - | - |

Table 11

## TABLE 12

### VOLATILE CONTENT AT 180°, 250°, AND 325°F WITH AGING

| Run No. | 1 | | | 2 | | | 3 | | |
|---|---|---|---|---|---|---|---|---|---|
| Lot No. | F-1070 | | | F-1070 | | | F-1070 | | |
| Process Variable | .35 BDMA | | | .55 BDMA | | | .75 BDMA | | |
| | VOLATILES[1], % | | | | | | | | |
| Aging Time, Days | 180°F | 250°F | 325°F | 180°F | 250°F | 325°F | 180°F | 250°F | 325°F |
| 0 | - | - | 1.44 | - | - | 1.26 | - | - | 1.01 |
| 1 | .52 | 1.66 | 2.03 | .47 | 1.39 | 1.43 | .43 | 1.05 | 1.07 |
| 2 | .45 | 1.33 | 1.82 | .42 | 1.25 | 1.60 | .40 | 0.99 | 1.20 |
| 3 | .59 | 1.44 | 1.68 | .51 | 1.28 | 1.40 | .43 | 0.83 | 0.97 |
| 4 | .56 | 1.39 | 1.92 | .43 | 1.23 | 1.67 | .37 | 1.09 | 1.34 |
| 5 | .55 | 1.33 | 2.03 | .45 | 1.23 | 1.71 | .45 | 1.04 | 1.32 |
| 6 | .75 | 1.51 | 2.13 | .73 | 1.48 | 1.95 | .81 | 1.14 | 1.21 |
| 7 | .44 | 1.59 | 2.00 | .38 | 1.31 | 1.62 | .45 | 1.48 | 1.70 |
| 8 | .57 | 1.57 | - | .39 | 1.44 | - | .35 | 1.12 | - |

(1) Data represents an average of three (3) test specimens.

No test was performed for blank spaces indicated.

Table 12

## TABLE 13

### A SUMMARY OF INCOMING QUALITY-CONTROL TEST DATA

| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A. Run No. | | | | | | | | | | | | | | | | |
| B. Lot No. | F-1070 | F-1070 | F-1070 | F-1082 | F-1082 | F-1121 | F-1102 | F-1102 | F-1102 | F-1118 | F-1108 | F-1108 | F-1118 | F-1154 | F-1154 | F-1155 |
| C. EMA Content, PHR | .35 | .55 | .75 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .45 |
| D. Special Gravity (Resin Bath) | .853 | .853 | .853 | .863 | .843 | .833 | .858 | .863 | .853 | .853 | .853 | .853 | .853 | .837 | .837 | .837 |
| E. Resin Bath Temp. °F | 75 | 75 | 75 | 75 | 75 | 75 | 65 | 75 | 75 | 75 | 75 | 75 | 75 | 75 | 75 | 75 |
| F. Running Speed, ft/min. | 65 | 65 | 65 | 65 | 65 | 65 | 65 | 55 | 75 | 83 | 65 | 75 | 65 | 80 | 65 | 65 |
| G. Tower Temp. (nominal), °F | 360 | 360 | 360 | 360 | 360 | 360 | 360 | 360 | 360 | 360 | 330 | 350 | 310 | 360 | 345 | 350 |
| H. Average Measured Temp, °F | 363 | 357 | 354 | 367 | 364 | 363 | 345 | 345 | 368 | 361 | 390 | 391 | 347 | 368 | 345 | 350 |
| I. Process Variable | C | (Nominal) | C | D | D | D | E | F | F | F | G | F G | G | F F | F G | G G |
| **J. PREPREG PROPERTY** | | | | | | | | | | | | | | | | |
| 1. Volatile, % | 1.44 | 1.26 | 1.01 | 1.40 | 1.26 | 0.94 | 1.33 | 1.26 | 1.28 | 1.58 | 1.22 | 1.45 | 1.33 | 1.27 | 1.22 | 1.19 |
| 2. Resin Content, % | 21.2 | 21.0 | 20.3 | 25.3 | 20.4 | 17.7 | 27.1 | 19.5 | 21.5 | 23.7 | 21.5 | 21.6 | 23.3 | 17.6 | 17.7 | 17.4 |
| 3. Resin Flow, % | 10.3 | 10.0 | 8.9 | 13.3 | 8.1 | 4.4 | 10.2 | 7.2 | 9.6 | 12.6 | 10.2 | 13.3 | 29.0 | 7.1 | | 7.5 |
| 4. Gel Time, Sec | 193 | 144 | 110 | 134 | 126 | 155 | 144 | 105 | 120 | 177 | 125 | 152 | 257 | 175 | 20 | 201 |
| 5. Viscosity, Centistokes | 1.736 | 1.776 | 1.780 | 1.935 | 1.065 | - | 1.872 | 1.972 | 1.812 | 1.704 | 1.868 | 1.881 | 1.798 | 1.778 | 1.772 | 1.772 |
| 6. Strand Weight, gm/yd. | .632 | .632 | .633 | .634 | .637 | .644 | .631 | .632 | .630 | .629 | .639 | .636 | .633 | .642 | .641 | .645 |
| 7. Strand Stress, 10³ psi | 326.8 | 339.0 | 368.4 | 371.3 | 345.0 | 319.4 | 350.6 | 348.7 | 348.5 | 371.7 | 367.5 | 367.5 | 362.2 | 336.4 | 339.0 | 312.6 |
| 8. MDL Composite Stress, 10³ psi | 323.9 | 317.9 | 215.9 | 193.1 | 210.4 | 227.2 | 219.1 | 226.2 | 223.0 | - | 210.2 | 223.5 | - | 221.0 | 225.3 | 223.5 |
| 9. MDL Fiber Stress, 10³ psi | 356.0 | 352.5 | 347.0 | 330.4 | 336.0 | 336.6 | 348.5 | 348.2 | 358.9 | - | 328.6 | 363.9 | - | 351.4 | 352.3 | 347.9 |
| 10. Band Width (Lot), 10⁻³ in. | - | - | - | - | - | - | - | - | - | 89 | - | - | - | - | - | - |
| 11. Band Width (Roll), 10⁻³ in. | 77 | 67 | 66 | 71 | 68 | - | - | 69 | 71 | - | 73 | 63 | 71 | 71 | 75 | 72 |

Table 13

TABLE 14

A SUMMARY OF OUTGOING QUALITY CONTROL TEST DATA

| Run No. | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lot No. | P-1070 | P-1070 | P-1070 | P-1082 | P-1082 | P-1122 | P-1102 | P-1102 | P-1102 | P-1118 | P-1108 | P-1108 | P-1118 | P-1154 | P-1154 | P-1155 |
| HMA Content, phr | .35 | .55 | .75 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .55 | .45 |
| Virgin Gel Time, sec | 222 | 163 | 128 | 158 | 158 | 153 | 164 | 164 | 164 | 154 | 155 | 155 | 154 | 167 | 167 | 240 |
| Sol'n Gel Time | 222 | 154 | 104 | 154 | 133 | 148 | 135 | 139 | 139 | 137 | 148 | 135 | 127 | 175 | 195 | 194 |
| Sol'n Viscosity, Centistokes | .6863 | .6389 | .6654 | .7252 | .6267 | .6501 | .6615 | .6503 | .6351 | .7052 | .6400 | .6700 | .6987 | .6000 | .5975 | .6192 |
| Running Speed, ft/min | 65 | 65 | 65 | 65 | 65 | 65 | 65 | 55 | 75 | 83 | 65 | 75 | 65 | 80 | 65 | 65 |



Table 14

## TABLE 15

### RESIN CONTENT WITH VARIOUS SPECIFIC GRAVITIES

### AND RUNNING SPEEDS

| Run No. | Specific Gravity at 75°F | Running Speed, ft/min | No. of Rolls Tested | Resin Content, % Av ($\bar{X}$) | Std Deviation |
|---|---|---|---|---|---|
| 6 | .833 | 65 | 4 | 17.66 | 0.31 |
| 15 | .837 | 65 | 4 | 17.76 | 0.70 |
| 5 | .843 | 65 | 6 | 20.33 | 0.75 |
| 1 | .853 | 65 | 6 | 21.36 | 0.33 |
| 2 | .853 | 65 | 6 | 21.06 | 0.57 |
| 3 | .853 | 65 | 6 | 20.81 | 0.54 |
| 11 | .853 | 65 | 6 | 21.80 | 0.51 |
| 12 | .853 | 65 | 6 | 21.81 | 0.20 |
| 4 | .863 | 65 | 6 | 25.33 | 0.63 |
| 8 | .853 | 55 | 6 | 19.40 | 0.51 |
| 9 | .853 | 75 | 6 | 21.48 | 0.22 |
| 10(1) | .853 | 83 | 3 | 23.80 | 0.58 |
| 17 | .853 | 65 | 5 | 22.10 | 0.48 |
|  |  |  | 5(3) | 22.43 | 0.22 |
|  |  |  | 5(3) | 22.34 | 0.22 |
| 18(2) | .853 | 65 | 6 | 22.05 | 0.34 |

(1) These rollers in resin bath.    (2) Pre-wet roller.    (3) Test in five areas within a roll.

Table 15

## TABLE 16

## VISCOSITY AND GEL TIME WITH VARIOUS RUNNING SPEEDS

### AND TOWER TEMPERATURES

| Run No.(1) | Running Speed, (ft/min) | Tower Temp. °F | Viscosity, Centistoke/sec | | | Gel Time/sec | | |
|---|---|---|---|---|---|---|---|---|
| | | | No. of Rolls Tested | Av (X̄) | Std. Deviation | No. of Rolls Tested | Av (X̄) | Std. Deviation |
| 8 | 55 | 365 | 5 | 1.972 | 0.045 | 10 | 105 | 2 |
| 2 | 65 | 357 | 6 | 1.776 | 0.024 | 9 | 144 | 5 |
| 4 | 65 | 367 | 10 | 1.835 | 0.049 | 9 | 134 | 4 |
| 5 | 65 | 364 | 9 | 1.865 | 0.025 | 9 | 126 | 5 |
| 7 | 65 | 365 | 5 | 1.872 | 0.063 | 5 | 144 | 10 |
| 9 | 75 | 368 | 4 | 1.812 | 0.008 | 10 | 120 | 1 |
| 14 | 80 | 368 | 6 | 1.778 | 0.033 | 6 | 195 | 6 |
| 10 | 83 | 361 | 5 | 1.704 | 0.013 | 4 | 177 | 7 |
| 13 | 65 | 347 | 5 | 1.758 | 0.023 | 4 | 167 | 3 |
| 15 | 65 | 345 | 6 | 1.794 | 0.016 | 6 | 190 | 4 |
| 11 | 65 | 390 | 4 | 1.968 | 0.032 | 5 | 125 | 9 |
| 12 | 75 | 391 | 5 | 1.881 | 0.042 | 5 | 162 | 9 |

(1) Resin mixes used for these runs all contained 0.55 phr EDMA.

Table 16

TABLE 17

## GEL TIME AND VISCOSITY WITH VARIOUS AMOUNTS OF BDMA

| Run No. | BDMA Content (phr) | Virgin Gel Time (sec) | Tower Temp. (°F) | Gel Time, Sec. No. of Rolls Tested | Gel Time, Sec. Av (X) | Gel Time, Sec. Std. Deviation | Viscosity, Centistoke/sec No. of Rolls Tested | Viscosity, Centistoke/sec Av (X) | Viscosity, Centistoke/sec Std. Deviation |
|---|---|---|---|---|---|---|---|---|---|
| 1 | .35 | 222 | 363 | 10 | 193 | 3 | 8 | 1.736 | 0.014 |
| 16 | .45 | | | 6 | 200 | 3 | 6 | 1.791 | 0.014 |
| 2 | .55 | 163 | 357 | 9 | 144 | 5 | 6 | 1.776 | 0.024 |
| 4 | .55 | 158 | 367 | 9 | 134 | 4 | 10 | 1.835 | 0.049 |
| 5 | .55 | 158 | 364 | 9 | 126 | 5 | 9 | 1.865 | 0.025 |
| 7 | .55 | 164 | 365 | 5 | 144 | 10 | 5 | 1.872 | 0.063 |
| 3 | .75 | 128 | 354 | 10 | 110 | 4 | 8 | 1.780 | 0.020 |

Table 17

## TABLE 18

### TEST DATA ON RESIN CONTENT CONTROL RUNS

| | | |
|---|---|---|
| Run No. | 17 | 18 |
| Lot No. | F-118 | F-1121 |
| Control Device | 3 rollers | Pre-wet roller |
| BDMA Content, phr | .55 | .55 |
| Specific Gravity (Resin Bath) | .853 | .853 |
| Resin Bath Temp. °F | 75 | 75 |
| Running Speed, ft/min | 65 | 65 |
| Tower Temp. | 360 | 360 |

**Prepreg Property**

| | | | |
|---|---|---|---|
| 1 | Volatile, % | 1.48 | 0.99 |
| 2 | Resin Content (lot) % | 22.1 | 22.0 |
| 3 | Resin Content (roll) % | 22.4 | - |
| 4 | Resin Flow, % | 10.6 | 9.3 |
| 5 | Gel Time, sec | 156 | 141 |
| 6 | Viscosity, Centistokes | 1.820 | - |
| 7 | Strand Weight, gm/yd | .630 | .639 |
| 8 | Strand Stress, $10^3$ psi | 372.5 | 353.1 |

Table 18

## TABLE 19

### RESIN CONTENT AND TENSILE STRENGTH

### WITH VARIATIONS OF CREEL TENSION AND PACKAGE GEOMETRY

| Run No. | Process Variable | Resin Content (%) with Creel Tension (lb) | | | Strand Tension, (10³ psi) | | NOL Ring Fiber Stress (10³psi) | |
|---|---|---|---|---|---|---|---|---|
| | | 1.5 | 2.0 | 4.0 | 60 Waywind | 3 1/8 Waywind | 60 Waywind | 3 1/8 Waywind |
| 1 | .35 EDMA | 21.8 | 21.4 | 20.9 | 319.0 | 325.6 | 362.5 | 353.6 |
| 2 | .55 EDMA | 21.5 | 21.4 | 20.3 | 355.9 | 351.1 | 349.0 | 353.7 |
| 3 | .75 EDMA | 20.2 | 20.8 | 20.1 | 340.4 | 366.3 | 342.0 | 346.3 |
| 4 | .863 Sp. Gr. | 27.3 | 25.3 | 24.0 | 358.8 | 377.6 | 329.6 | 326.1 |
| 5 | .843 Sp. Gr. | 21.2 | 20.3 | 19.9 | 330.8 | 347.8 | 331.4 | 336.8 |
| 6 | .833 Sp. Gr. | 17.4 | 17.7 | 18.4 | 331.2 | 318.1 | 333.9 | 338.0 |
| 7 | 65°F Bath Temp. | 23.0 | 21.7 | 22.4 | 357.7 | 345.1 | 351.6 | 346.9 |
| 8 | 55 ft/min | 19.0 | 19.4 | 19.8 | 329.4 | 347.3 | 356.3 | 340.0 |
| 9 | 75 ft/min | 23.5 | 21.5 | 22.4 | 337.4 | 345.5 | 355.8 | 362.0 |
| 10 | 83 ft/min | 25.0 | 23.8 | 23.4 | 375.0 | 368.7 | - | - |
| 11 | 390°F T. Temp. | 25.2 | 21.8 | 21.0 | 372.0 | 364.7 | 330.2 | 327.0 |
| 12 | 390°F + 75 ft/min | 21.6 | 21.8 | 21.4 | 347.3 | 369.0 | 366.0 | 361.8 |
| 13 | 340°F T. Temp. | 24.6 | 23.4 | 23.0 | 386.9 | 380.3 | - | - |
| 14 | 80 ft/min + .837 Sp.Gr. | 18.9 | 17.6 | 16.5 | 313.9 | 340.4 | - | - |
| 15 | 345°F T.Temp.; .837 Sp. Gr. | 18.2 | 17.8 | 17.1 | 347.8 | 344.9 | - | - |
| 16 | .45 EDMA; 350°F T.Temp. | 17.0 | 15.1 | 15.3 | 316.2 | 310.7 | - | - |
| 17 | R.C. control (3 rollers) | 22.1 | 22.2 | 21.4 | - | 371.9 | - | - |
| 18 | R.C. control (Pre-wet roller) | 21.6 | 22.1 | 22.0 | 346.5 | 360.5 | - | - |

Table 19

## TABLE 20

MEASUREMENTS OF TOW TENSION AND RESIN CONTENT IN...

**Run No. 8**

**Lot No. F-1102**

Process Variables: 50 ft/min Admin, Speed

| Roll No. | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| **TENSION, lb.** | | | | | | | | | | |
| 1. Creel / 2. Location A | 2.0 4.5 | 2.0 4.5 | 2.0 4.8 | 2.0 4.5 | 2.0 5.0 | 2.0 5.0 | 1.5 4.0 | 1.5 5.0 | 4.0 5.5 | 4.0 5.0 |
| 3. Location B | 10.0 | 10.0 | 9.8 | 10.0 | 10.0 | 11.0 | 6.3 | 6.0 | 4.5 | 3.5 |
| 4. Location C | 10.0 17.0 | 10.0 17.0 | 10.0 17.0 | 10.0 15.0 | 7.5 17.5 | 9.0 17.0 | 9.0 16.0 | 6.8 14.0 | 10.0 14.0 | 13.8 10.0 |
| 5. Location D | 1.0 3.0 | 12.0 3.5 | 12.0 3.5 | 13.0 3.5 | 13.0 2.5 | 12.0 4.0 | 13.0 3.0 | 12.0 3.0 | 15.0 3.0 | 10.0 16.8 |
| | 4.3 | 3.0 | 2.8 | 3.0 | 2.5 | 2.5 | 3.0 | 3.0 | 2.5 | 2.5 3.5 |
| **RESIN CONTENT, %** | | | | | | | | | | |
| ACC Data | 19.5 | 19.3 | 19.7 | 16.5 | 19.2 | 20.2 | 19.0 | 19.8 | 12.9 | -- |
| U.S.P. Data | 19.6 | 17.1 | 18.1 | 17.8 | 18.8 | 20.6 | 18.9 | 19.8 | 20.6 | -- |

**Run No. 2**

**Lot No. F-1102**

Process Variables: 75 ft/min Running Speed

| Roll No. | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|
| **TENSION, lb.** | | | | | | | | | | |
| 1. Creel | 2.0 | 5.0 | 2.0 | 2.0 | 2.0 | 2.0 | 1.5 | 1.5 | 4.0 | 4.0 |
| 2. Location A | 5.0 | 4.5 | 4.5 | 3.5 | 4.8 | 5.0 | 3.5 | 4.5 | 4.5 | 4.5 |
| 3. Location B | 10.0 | 10.0 | 10.5 | 15.5 | 14.8 | 13.5 | 15.0 | 17.0 | 17.0 | 14.5 |
| 4. Location C | 15.0 | 13.0 | 11.0 | 11.5 | 11.5 | 17.0 | 17.5 | 18.5 | 17.0 | 17.0 |
| 5. Location D | 5.0 | 1.5 | 5.0 | 5.0 | 5.0 | 4.5 | 4.5 | 4.8 | 4.8 | 5.0 |
| **RESIN CONTENT, %** | | | | | | | | | | |
| ACC Data | 21.4 | 21.6 | 21.6 | 21.2 | 21.3 | 21.8 | 23.2 | 23.9 | 23.3 | 21.7 |
| U.S.P. Data | 22.2 | 21.6 | 21.6 | 21.2 | 21.9 | 22.2 | 18.7 | 22.0 | 21.8 | 21.9 |

*Location of measurements is shown in Figure 2.

Table 20

TABLE 21

**BAND-WIDTH MEASUREMENTS**

| Run No. | Lot No. | Process Variable | Band Width, $10^{-3}$ inch | | | | |
|---|---|---|---|---|---|---|---|
| | | | No. Tension | 10 lb Loc. 1 | Weight Loc. 2 | Tension Loc. 3 | Edge |
| 1 | F-1070 | 0.35 EDMA | 85 | 77 | 73 | 73 | 106 |
| 2 | F-1070 | Nominal (0.55) | 76 | 76 | 67 | 67 | 92 |
| 3 | F-1070 | 0.75 EDMA | 76 | 66 | 65 | 66 | 118 |
| 4 | F-1082 | 0.863 sp. gr. | 77 | 76 | 70 | 67 | 96 |
| 5 | F-1082 | 0.843 sp. gr. | 72 | 71 | 70 | 64 | 80 |
| 8 | F-1102 | 55 ft/min. | 74 | 64 | 71 | 72 | 97 |
| 9 | F-1102 | 75 ft/min | 76 | 76 | 69 | 68 | 97 |
| 10 | F-1118 | 83 ft/min | 100 | 98 | 89 | 81 | 116 |
| 11 | F-1108 | 390°F | 74 | 71 | 73 | 76 | 98 |
| 12 | F-1108 | 390°F + 75 ft/min | 84 | 79 | 80 | 91 | 101 |
| 13 | F-1118 | 340°F ft/min | 82 | 72 | 69 | 72 | 102 |
| 14 | F-1154 | 80 ft/min | 79 | 71 | 70 | 70 | 103 |
| 15 | F-1154 | 345°F | 84 | 70 | 76 | 79 | 109 |
| 16 | F-1155 | 0.45 EDMA + 350°F | 73 | 68 | 72 | 71 | 93 |
| 18 | F-1121 | R.C. Control | 76 | 78 | 72 | 75 | 100 |

Table 21

(A) RAW MATERIALS - BLENDING

(B) RAW GLASS - MELTING

(C) GLASS FEED - GLASS MARBLES

(D) GLASS MELT - MOULTEN GLASS

(E) GRAVITY EXTRUSION - "VIRGIN" FIBERS
(F) PROTECTIVE COATING APPLICATIONS - SIZING
(G) STRAND FORMING - GATHERING

STRAND
(H) STRAND ON FORMING TUBE

(I) OVEN BAKEOUT - SOLVENT REMOVABLE

(J) CREEL

STRANDS

(K) MULTIPLE END ROVING - GATHERING

(L) WINDING - ROVING PACKAGE

(M) PACKAGING AND SHIPMENT

FIBERGLASS MANUFACTURING PROCESS

A32 /:63-968

Figure 1

A270:63-903
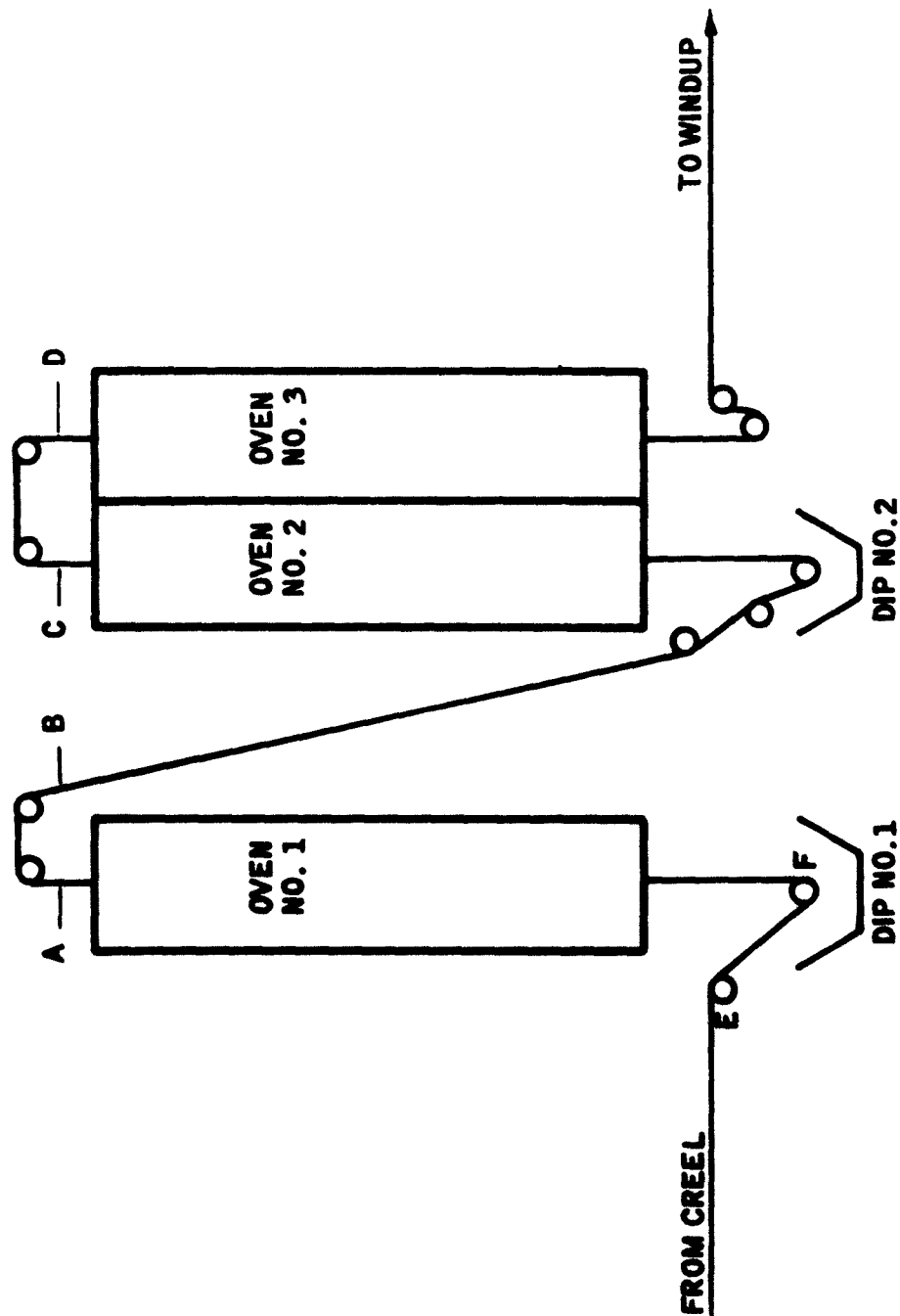
SCHEMATIC DIAGRAM OF U.S. POLYMERIC PREIMPREGNATION LINE

TO WINDUP

OVEN NO. 3

OVEN NO. 2

OVEN NO. 1

DIP NO.2

DIP NO.1

FROM CREEL

Figure 2

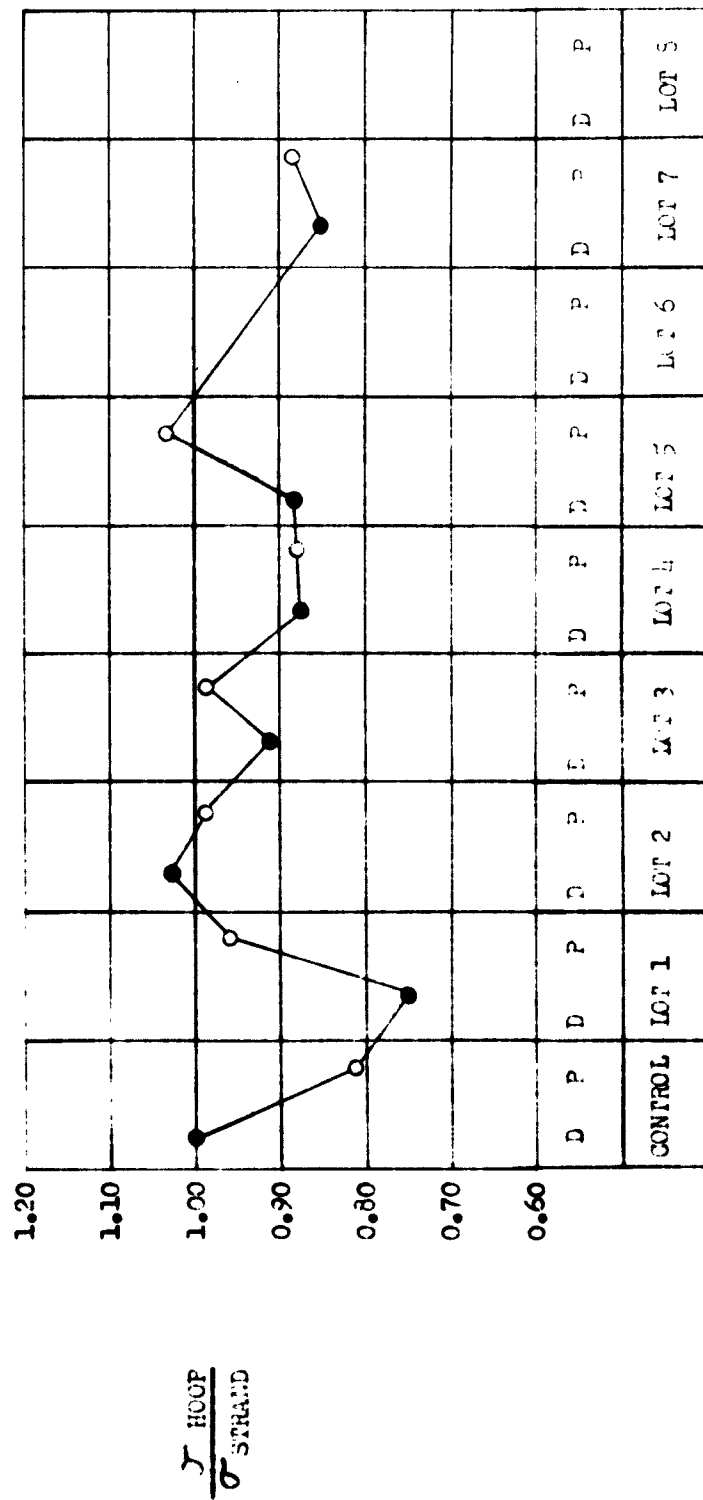COMPARISON OF NOL-RING DATA AND CHAMBER DATA
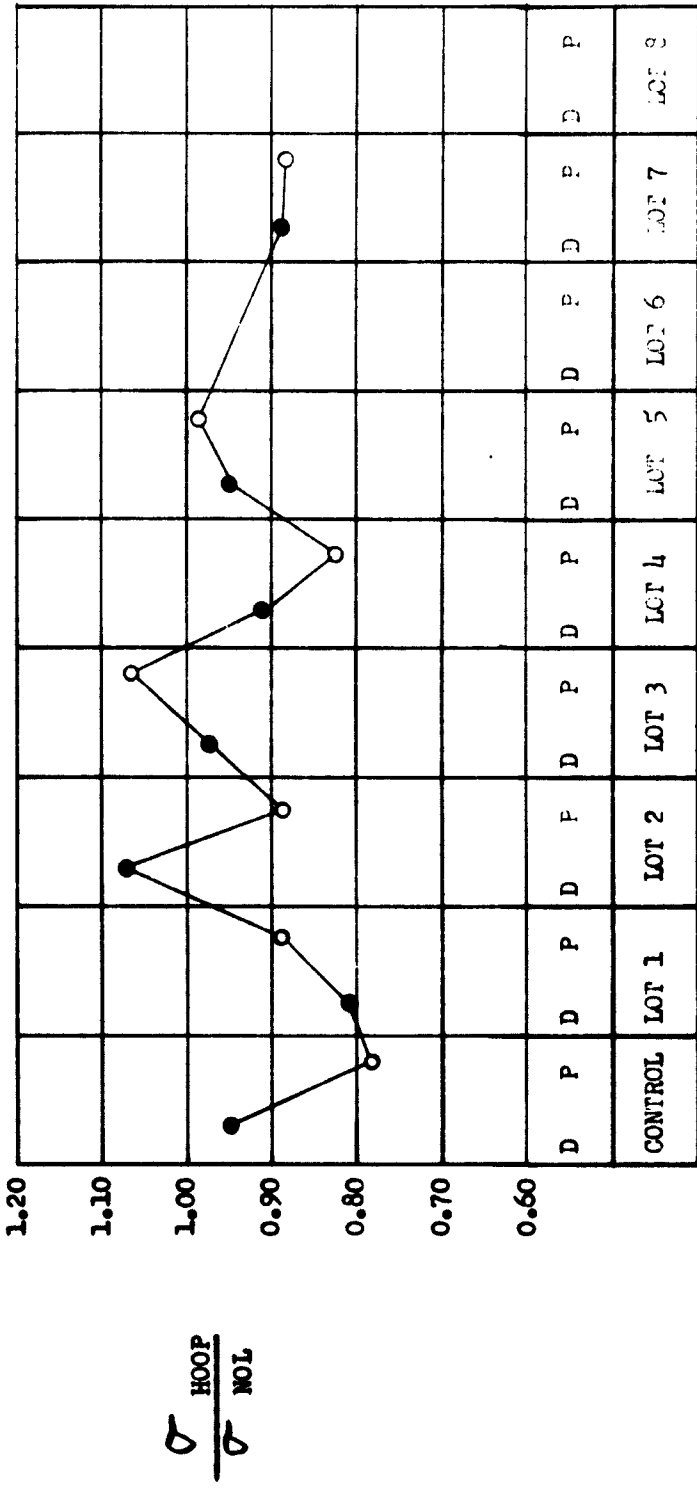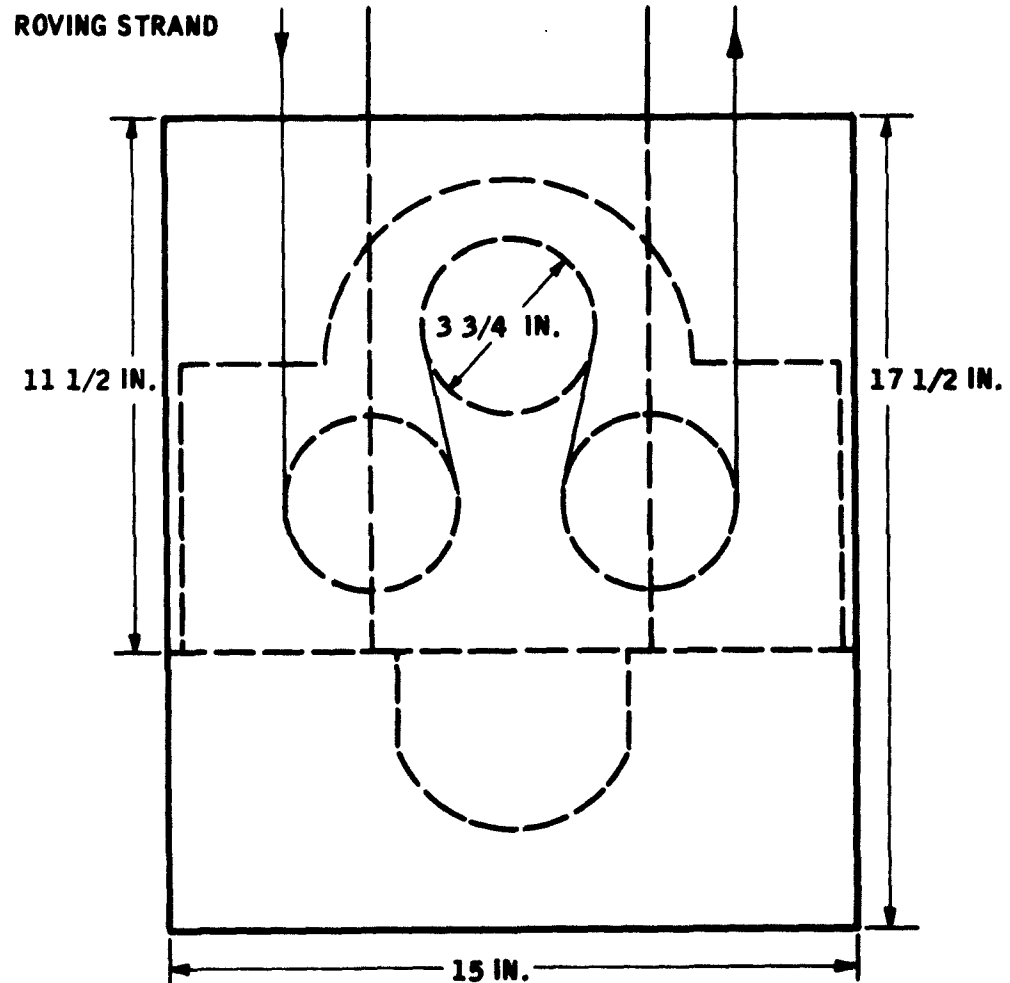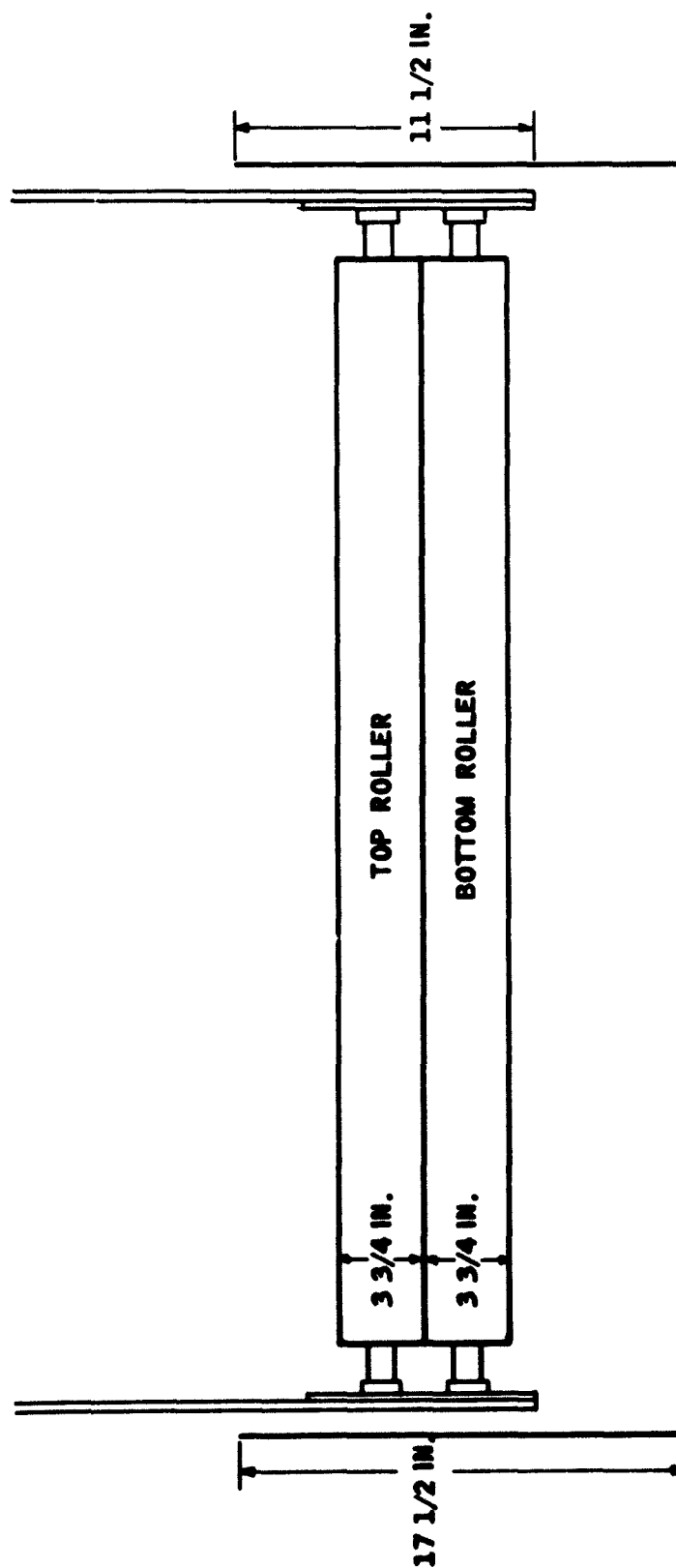
Figure 3

Figure 4

EQUIPMENT:

THREE 302/304 STAINLESS STEEL ROLLERS. LENGTH - 42 IN.   DIAMETER 3 3/4 IN.

SIX 302/304 STAINLESS STEEL END CAPS FOR ROLLERS

SIX BRONZE BUSHINGS TO FIT END CAPS

ROVING STRAND

11 1/2 IN.
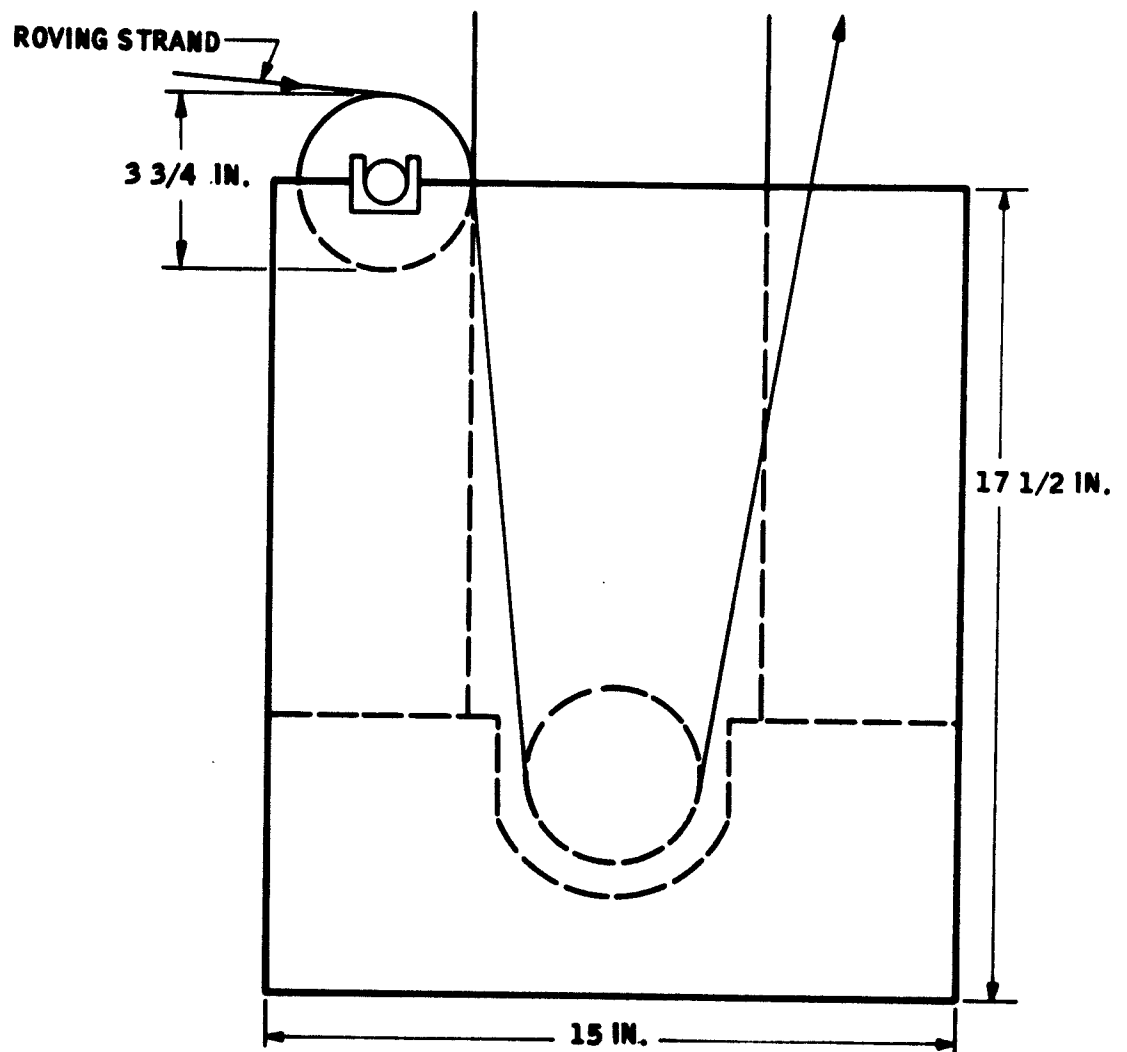
3 3/4 IN.

17 1/2 IN.

15 IN.

SKETCH  FOR  ROLLERS  IN  RESIN  BATH
(END  VIEW)

Figure 5

11 1/2 IN.

A270:63-855

TOP ROLLER

BOTTOM ROLLER

3 3/4 IN.

3 3/4 IN.

17 1/2 IN.

ROLLERS SHOWN WITH PAN SIDES AS REFERENCE
(SIDE VIEW)

Figure 6

ROVING STRAND

3 3/4 IN.

17 1/2 IN.

15 IN.

PAN WITH PRE-WET ROLLER
(END VIEW)

A270:63-857

Figure 7

A270:63-856

EQUIPMENT:
ONE ONE-FOOT 302/304 STAINLESS STEEL ROLLER, DIAMETER: 3 3/4 IN.

TWO 302/304 STAINLESS STEEL END CAPS FOR ROLLER

ONE FOUR-FOOT 302/304 STAINLESS STEEL SHAFT, DIAMETER 1 1/2 IN.

TWO TAILOR-MADE BRONZE BUSHINGS WITH SUPPORTS FOR BUSHINGS TO FIT THE ENDS OF THE SHAFT FOR ROTATION

BOTTOM ROLLER

12 IN.

49 3/4 IN.
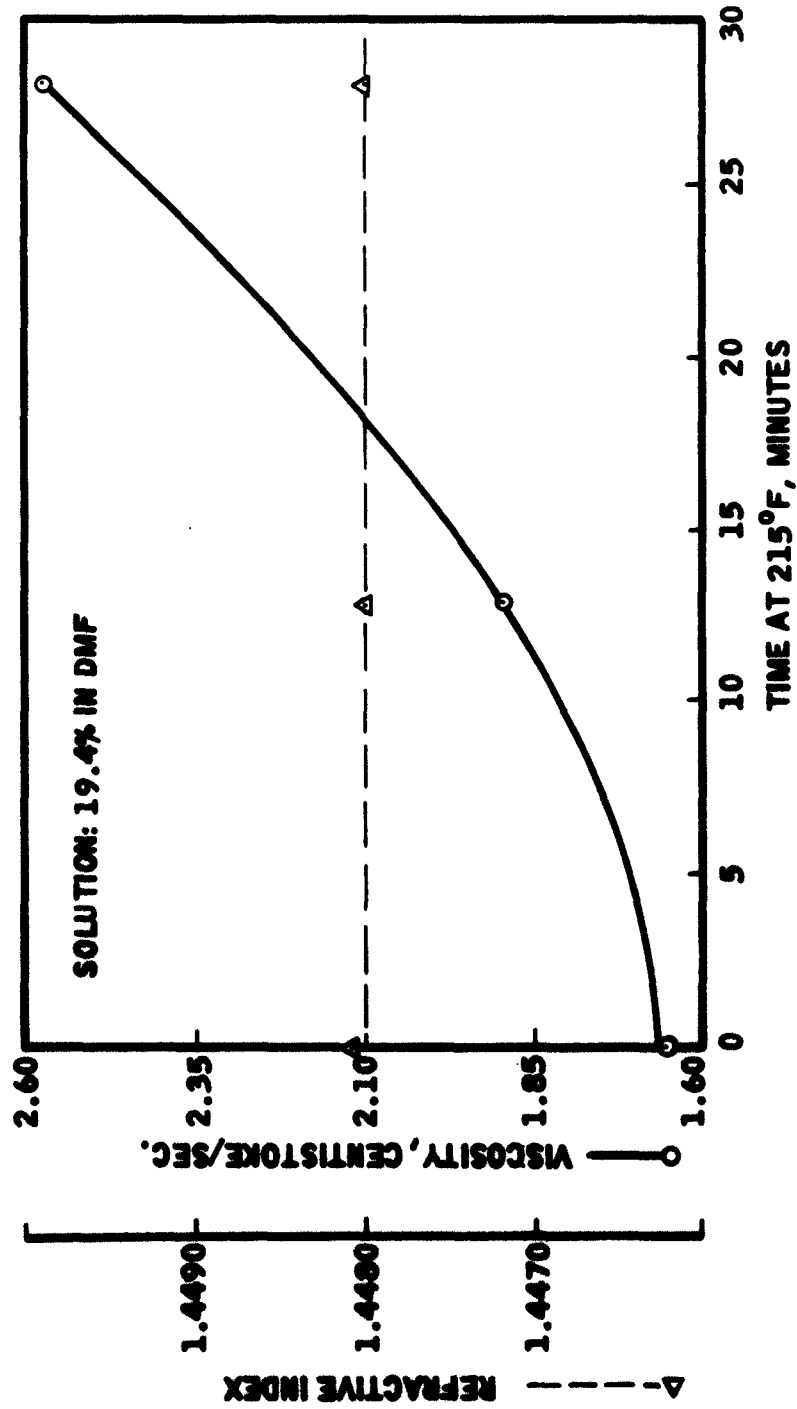
15 IN.
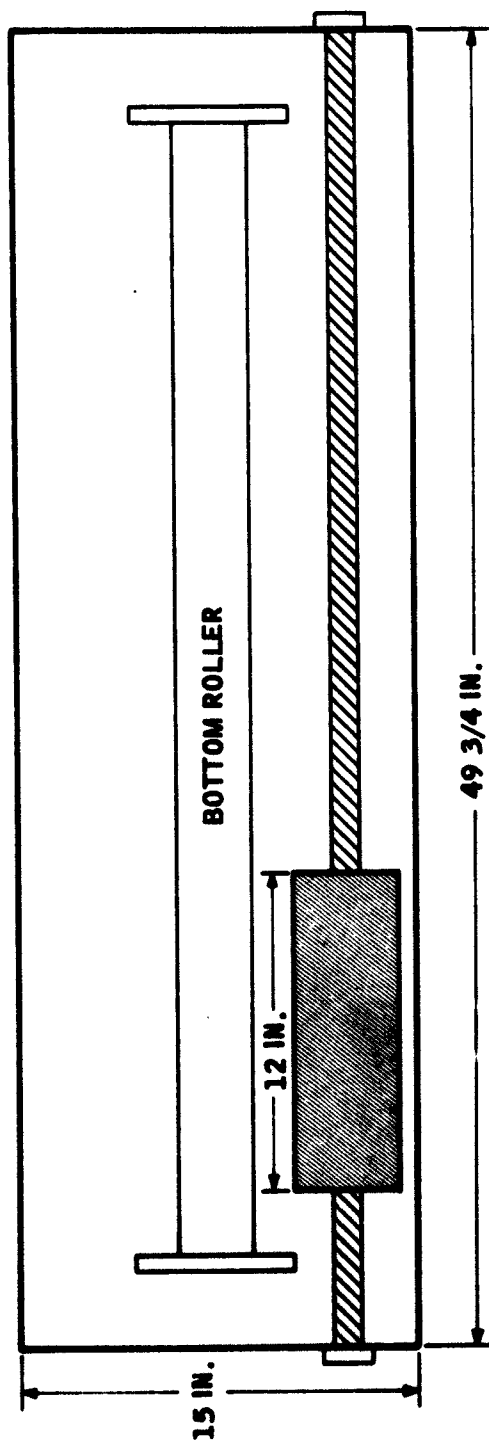
PAN WITH PRE-WET ROLLER
(TOP VIEW)

Figure 8

A270:63-852



EFFECT OF TIME AT 215°F ON VISCOSITY OF 58-68R/0.55 BDMA
RESIN SYSTEM

Figure 9

A270:63-851
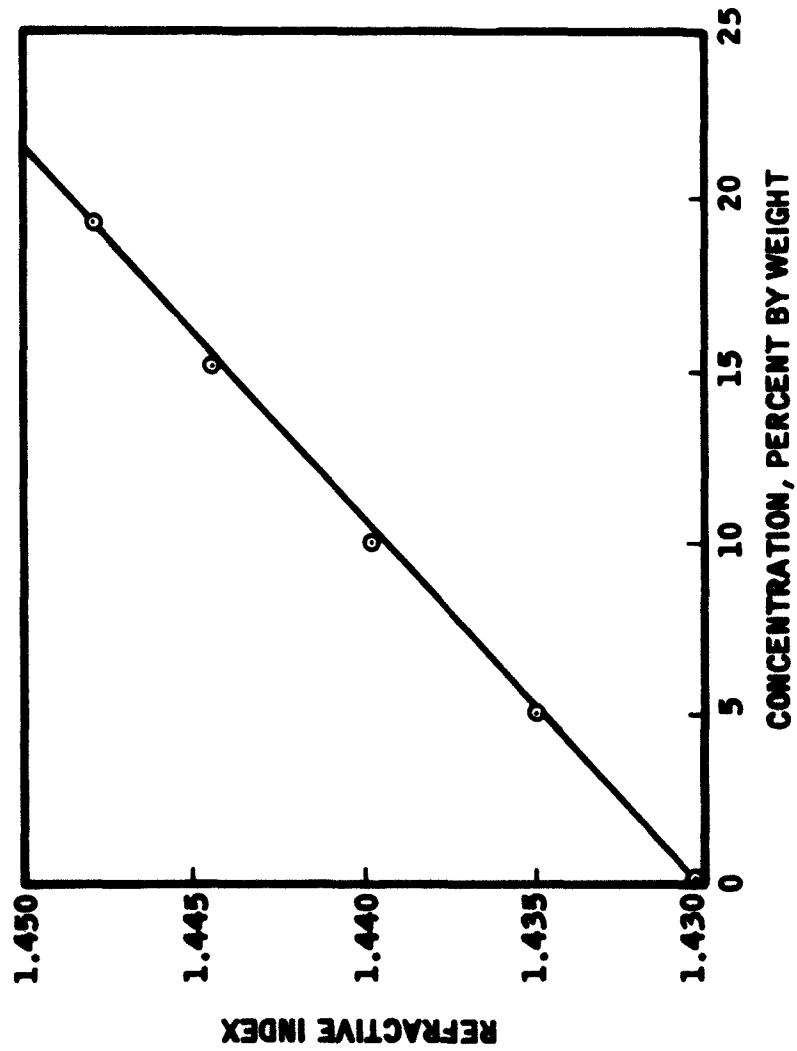


REFRACTIVE INDEX AS A FUNCTION OF CONCENTRATION OF
58-68R/0.55 BDMA RESIN SYSTEM IN DMF SOLVENT
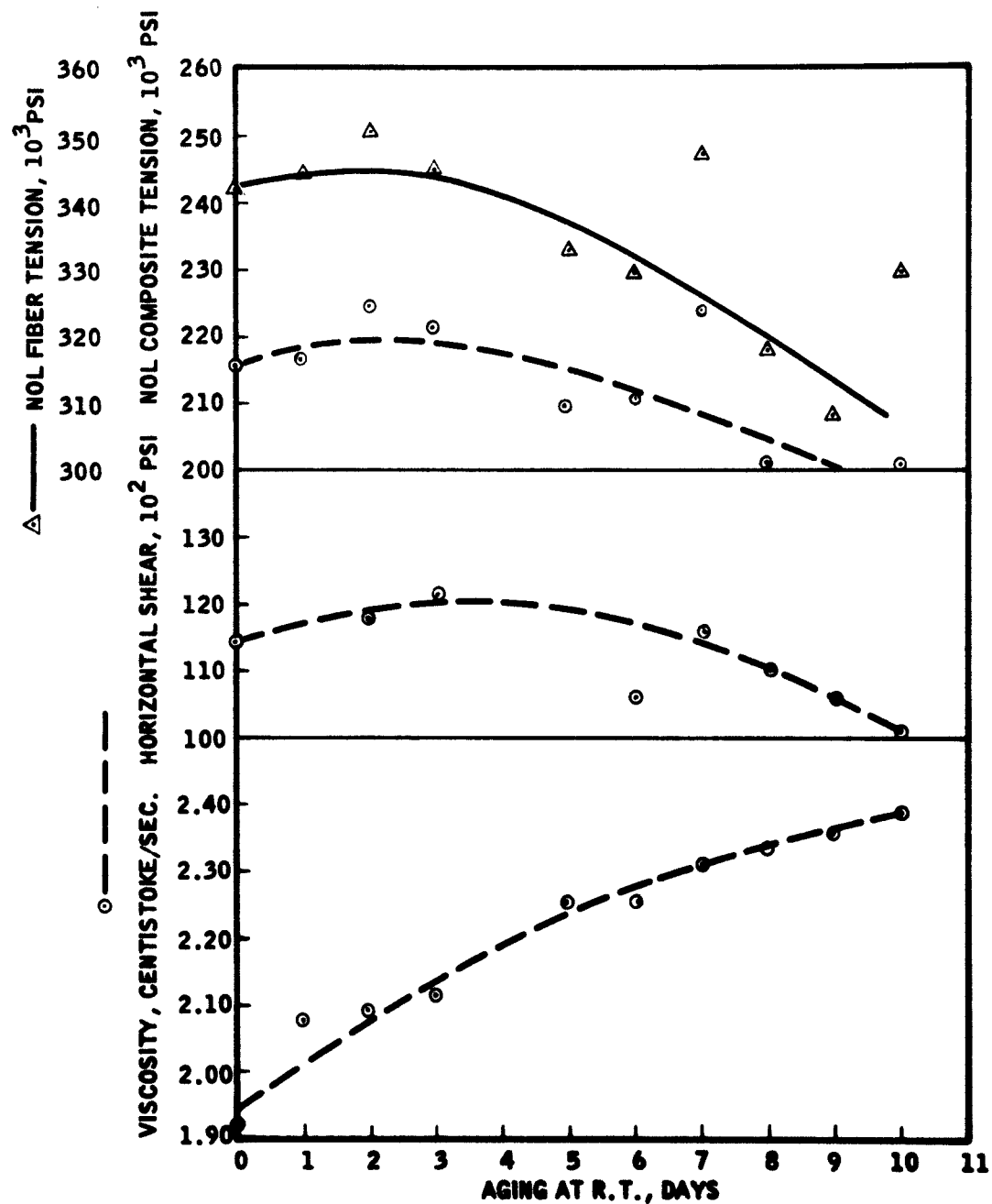
Figure 10

CORRELATION OF HORIZONTAL SHEAR AND NOL-RING
TENSILE STRENGTHS WITH VISCOSITY UPON AGING
(PROCESS VARIABLE: RUNNING SPEED AT 55 FT/MIN.)

A270:63-875
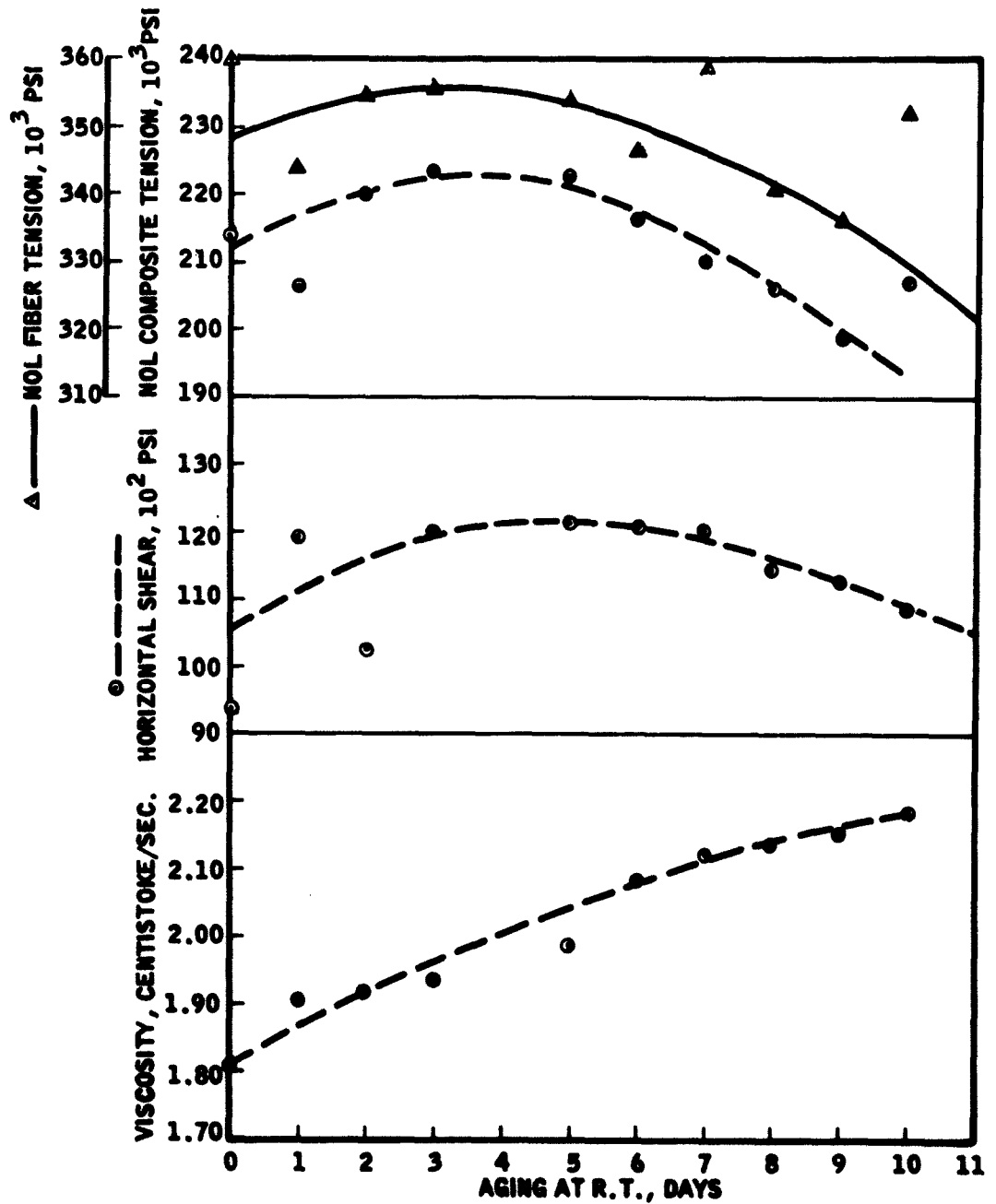
Figure 11

CORRELATION OF HORIZONTAL SHEAR AND NOL-RING
TENSILE STRENGTHS WITH VISCOSITY UPON AGING
(PROCESS VARIABLE: RUNNING SPEED AT 75 FT/MIN.)

A270:63-873

Figure 12

CORRELATION OF HORIZONTAL SHEAR AND NOL-RING
TENSILE STRENGTHS WITH VISCOSITY UPON AGING
(PROCESS VARIABLE: RUNNING SPEED AT 83 FT/MIN.)

A270:63-876

Figure 13

CORRELATION OF HORIZONTAL SHEAR AND NOL-RING
TENSILE STRENGTHS WITH VISCOSITY UPON AGING
(PROCESS VARIABLE: TOWER TEMPERATURE AT 340°F)

A270:63-877

Figure 14
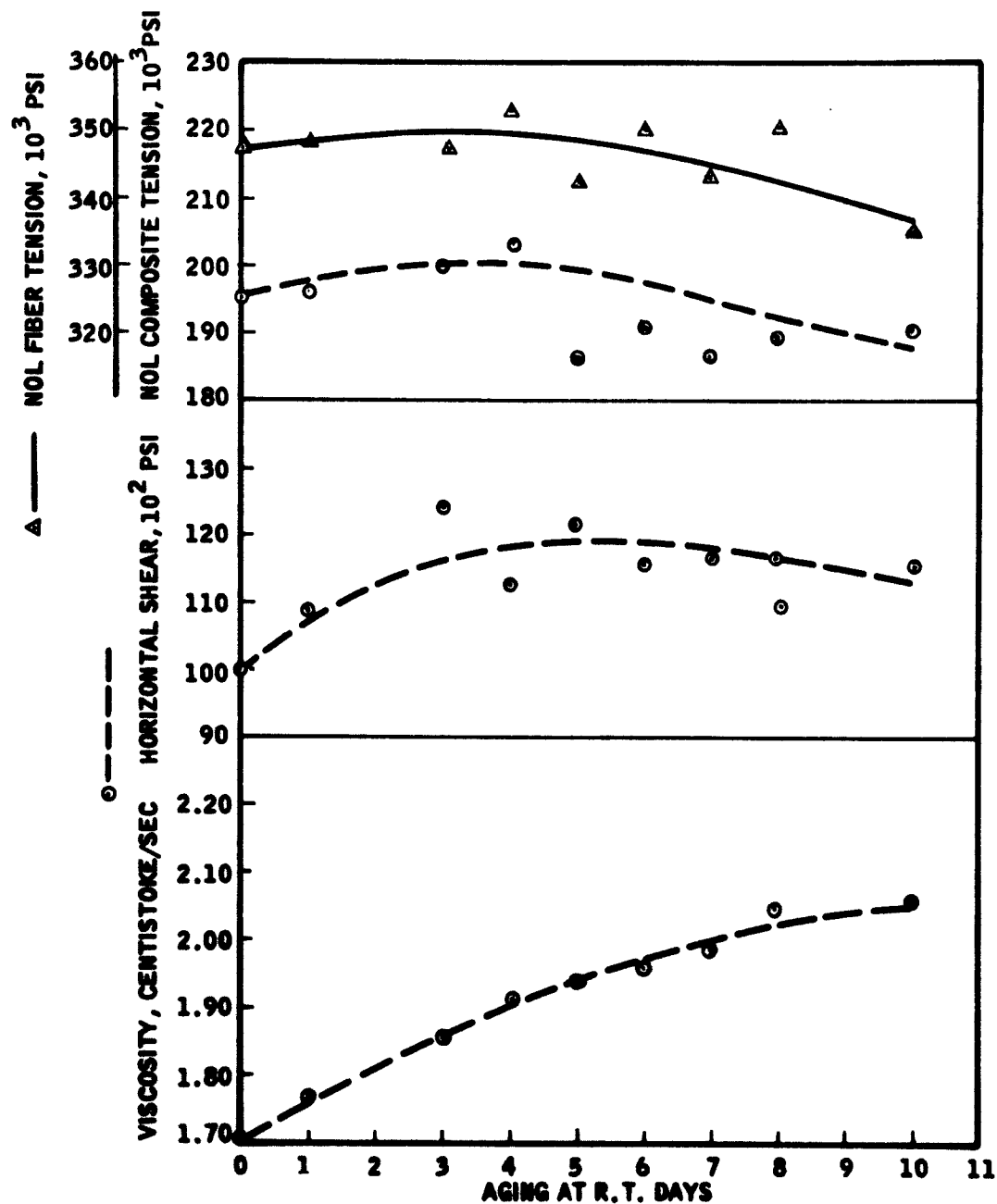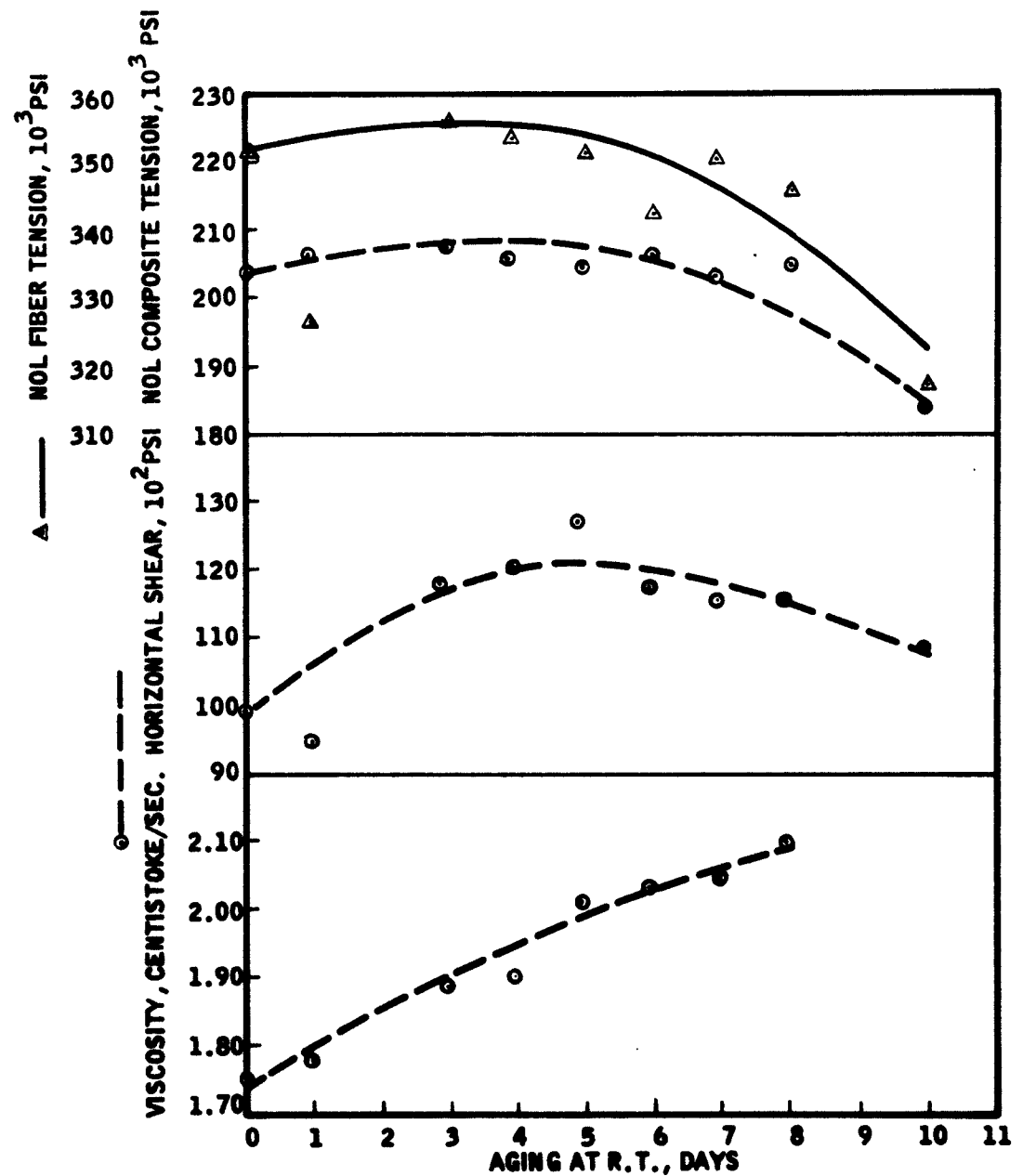
CORRELATION OF HORIZONTAL SHEAR AND NOL-RING
TENSILE STRENGTHS WITH VISCOSITY UPON AGING
(PROCESS VARIABLE: RUNNING SPEED AT 80 FT/MIN)

A270:63-873
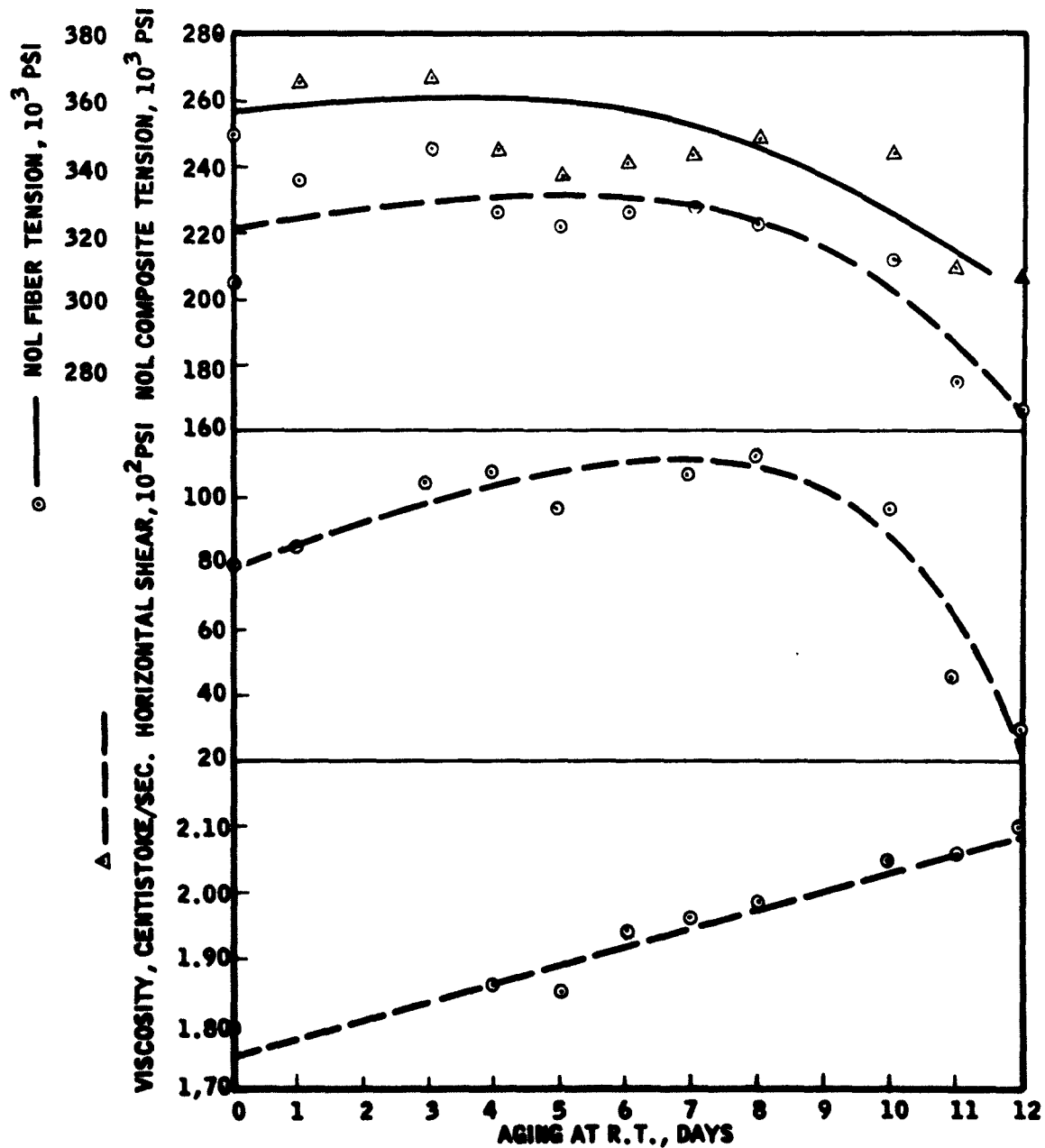
Figure 15

CORRELATION OF HORIZONTAL SHEAR AND NOL-RING
TENSILE STRENGTHS WITH VISCOSITY UPON AGING
(PROCESS VARIABLE: TOWER TEMPERATURE AT 345°F)

A270:63-878
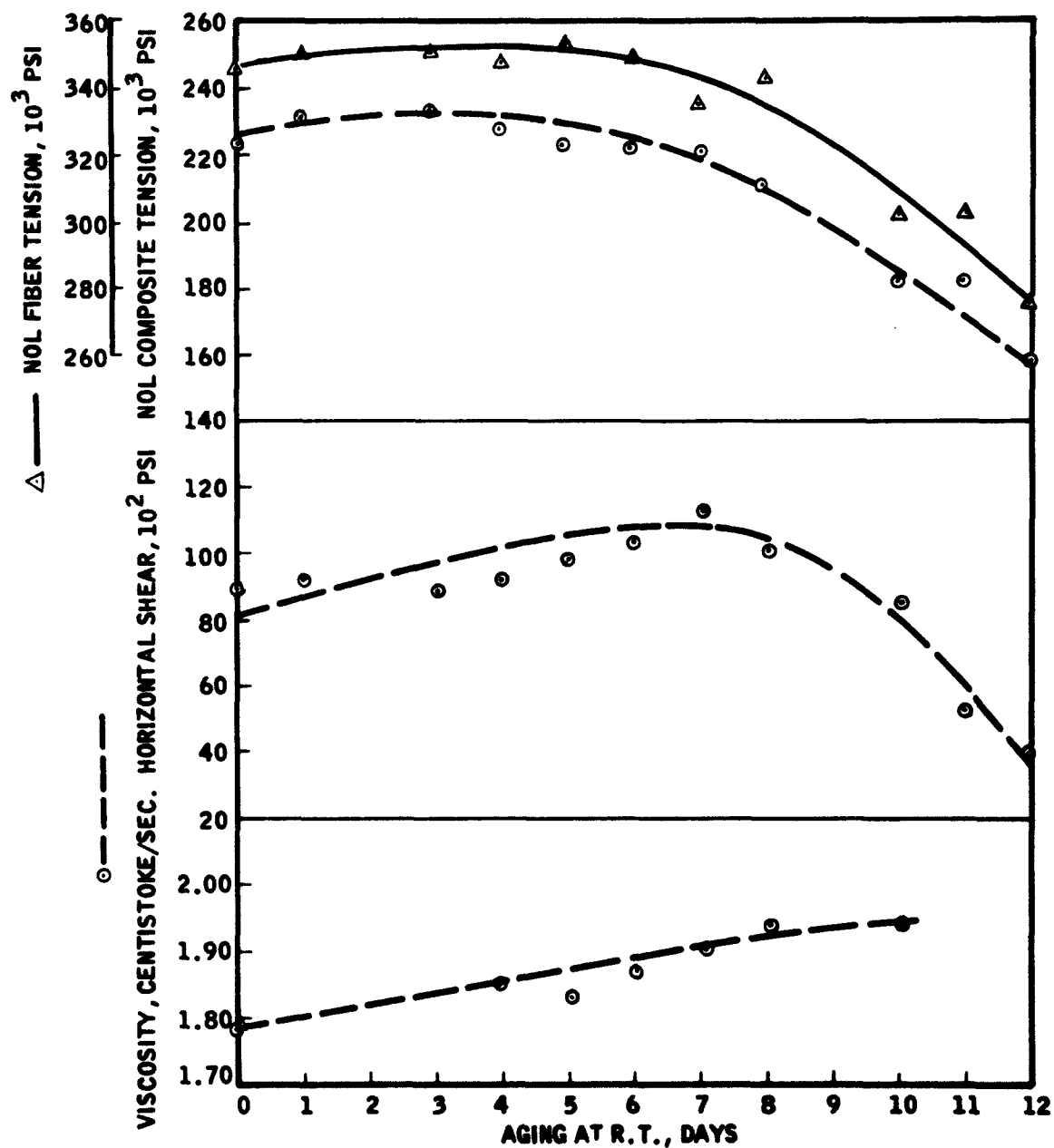
Figure 16

CORRELATION OF HORIZONTAL SHEAR AND NOL-RING
TENSILE STRENGTHS WITH VISCOSITY UPON AGING
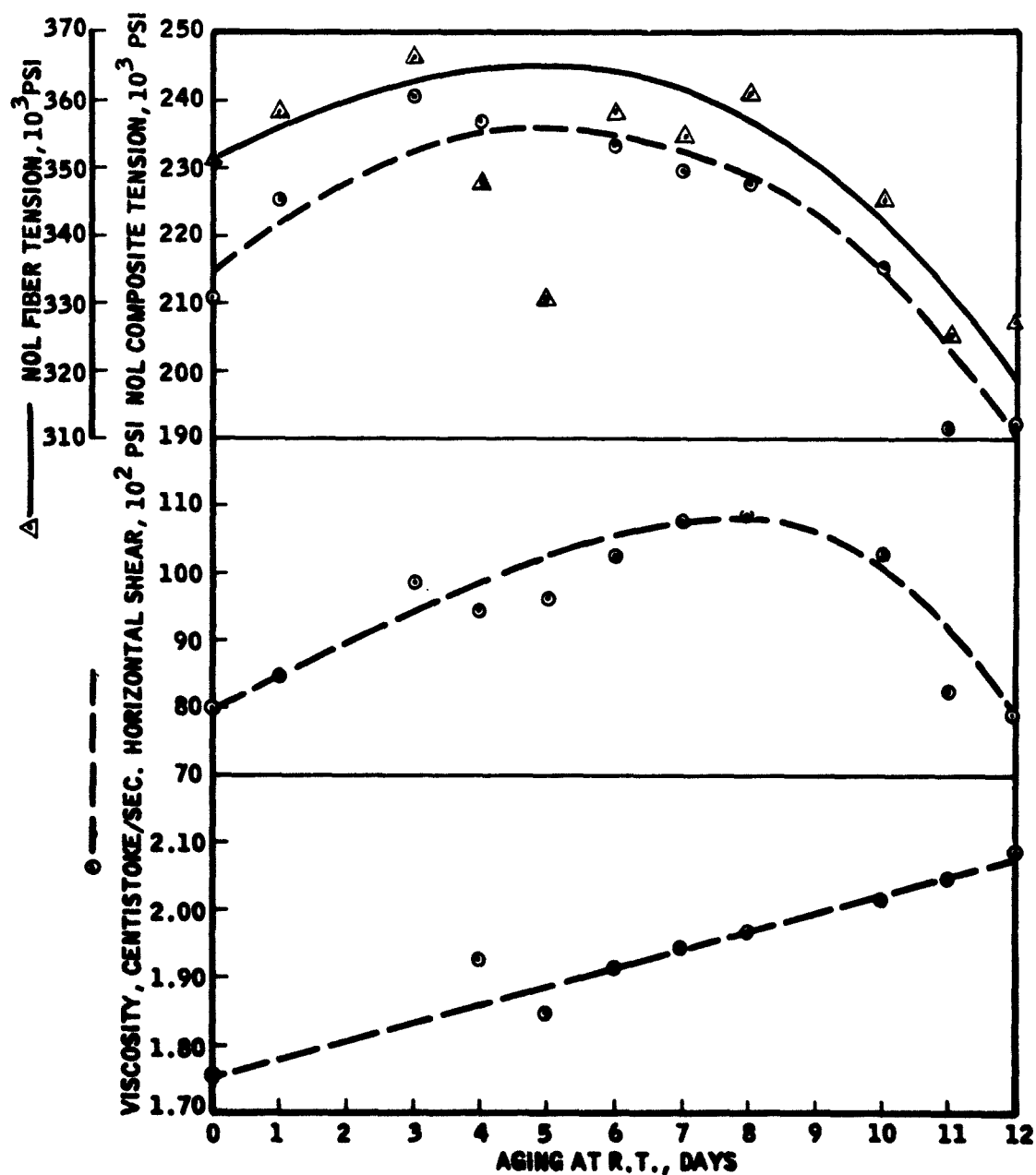(PROCESS VARIABLE = 0.45 BDMA CONTENT)

A270:63-885

Figure 17

MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA
REFRACTIVE INDEX OF 1.4480
IN DIMETHYL FORMAMIDE (DMF)
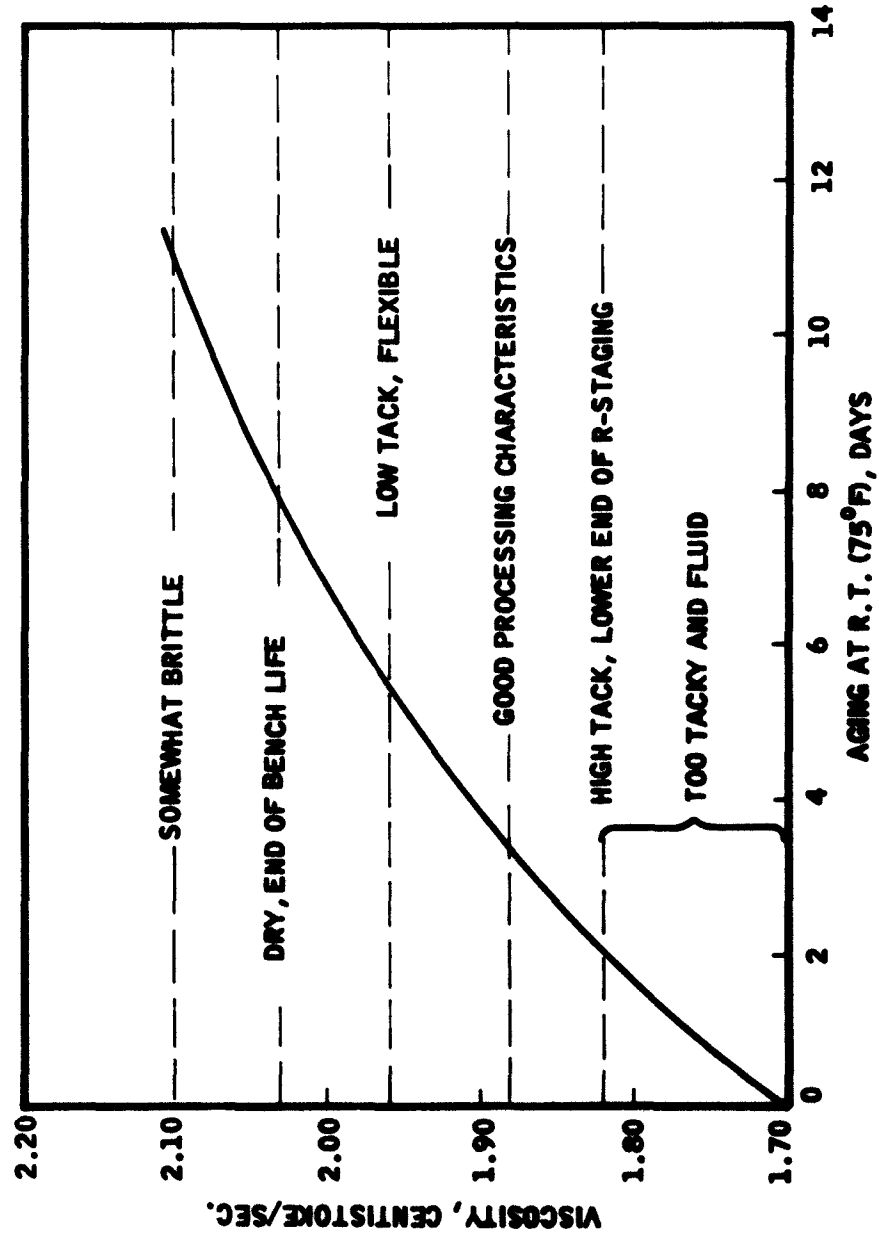
EFFECT OF AGING ON VISCOSITY

A270:63-850
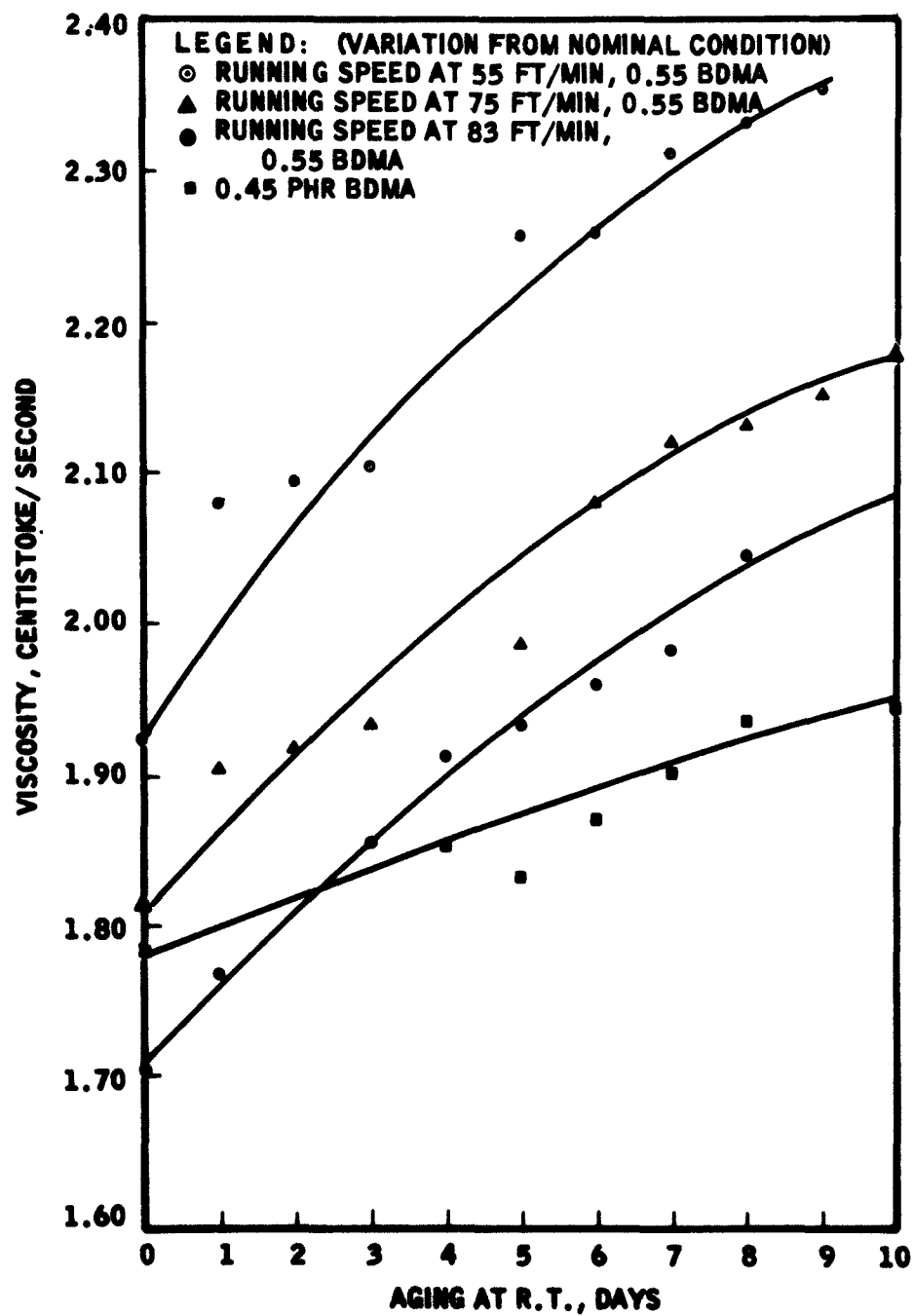
Figure 18

EFFECT OF AGING ON VISCOSITY FOR PREPREGS

PRODUCED UNDER VARIOUS PROCESSING CONDITIONS

A270:63-884

Figure 19

A270:63-879



MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0-55 PHR BDNA

PROCESS CONDITIONS: TOWER TEMPERATURE AT 360°F
RUNNING SPEED AT 65 FT/MIN.

RESIN CONTENT, PERCENT

RESIN BATH SPECIFIC GRAVITY AT 75°F
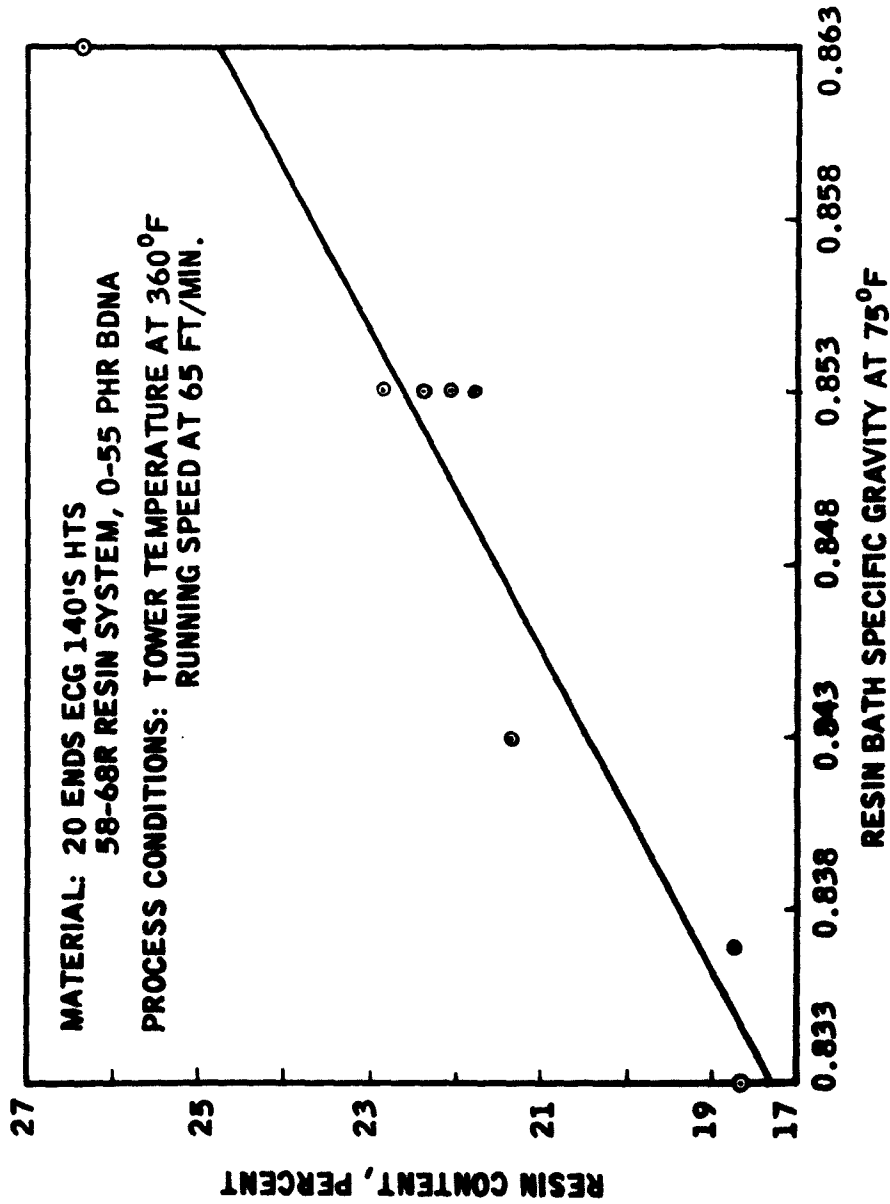
EFFECT OF SPECIFIC GRAVITY OF THE RESIN IMPREGNATING
BATH UPON RESIN CONTENT

Figure 20

A270:63-880



MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

PROCESS CONDITIONS: TOWER TEMPERATURE AT 360°F
RESIN SPECIFIC GRAVITY OF 0.853
AT 75°F.

RESIN CONTENT, PERCENT
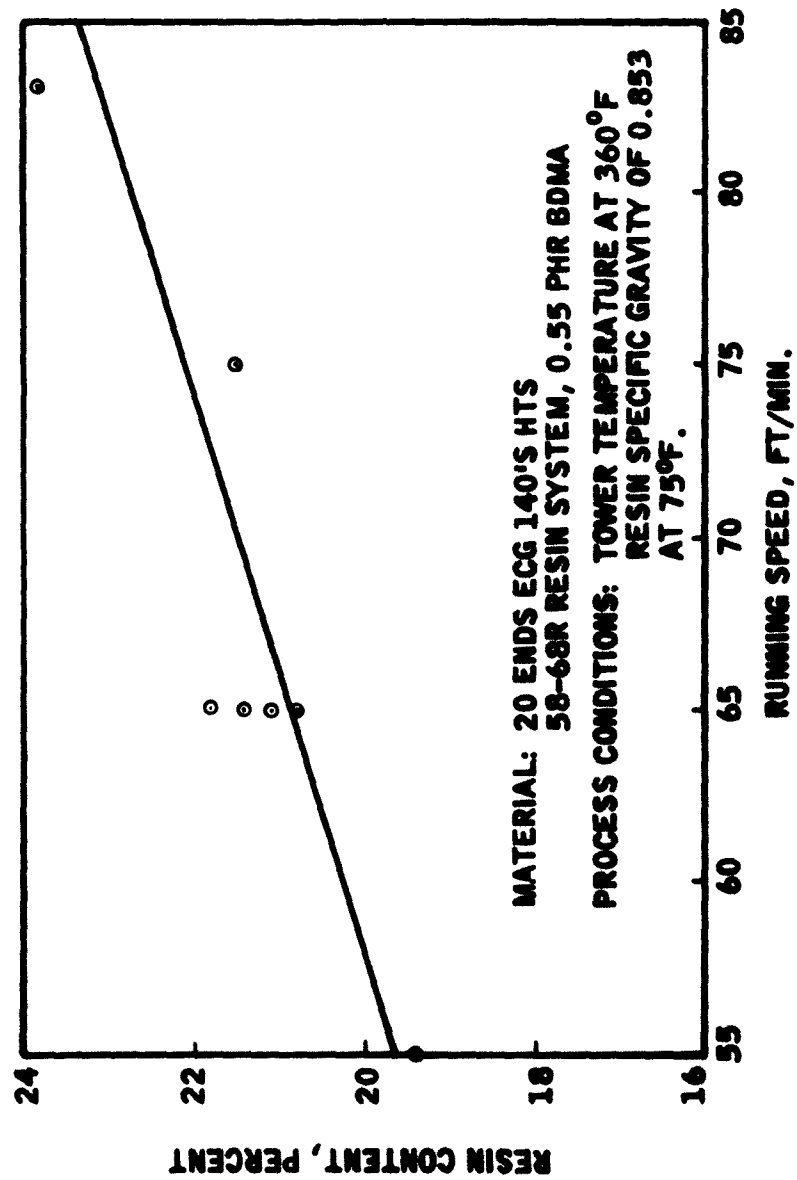
RUNNING SPEED, FT/MIN.

EFFECT OF RUNNING SPEED ON RESIN CONTENT
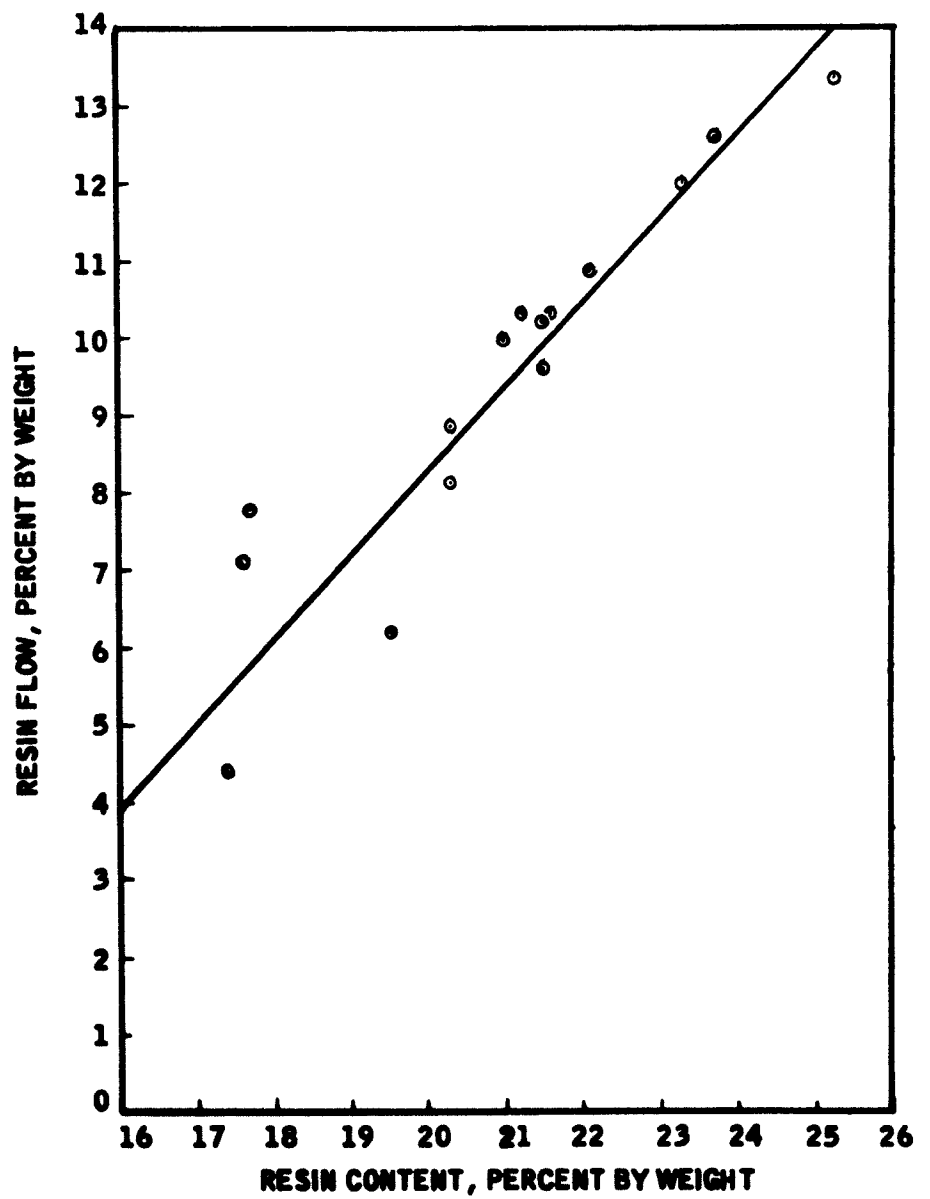
Figure 21

CORRELATION OF RESIN CONTENT AND RESIN FLOW

A270:63-883

Figure 22

A270:63-882

MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

PROCESS CONDITIONS: RUNNING SPEED AT 65 FT/MIN.
RESIN SPECIFIC GRAVITY OF 0.853 AT 75°F
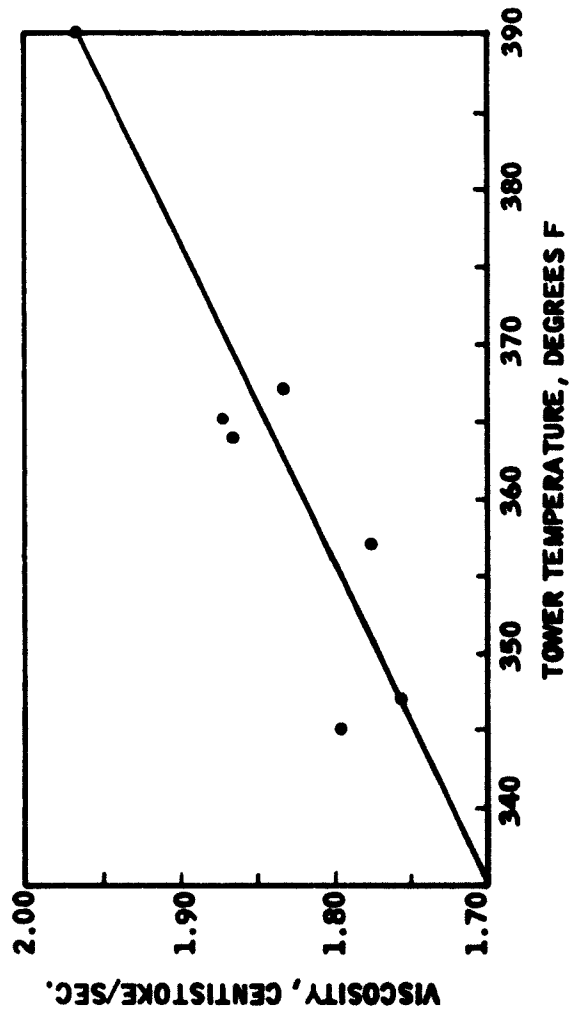


EFFECT OF TOWER TEMPERATURE ON VISCOSITY

Figure 23

A270:63-881

MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

PROCESS CONDITIONS: TOWER TEMPERATURE AT 365°F
RESIN SPECIFIC GRAVITY OF 0.853 AT 75°F

EFFECT OF RUNNING SPEED ON VISCOSITY

Figure 24

A270:63-898
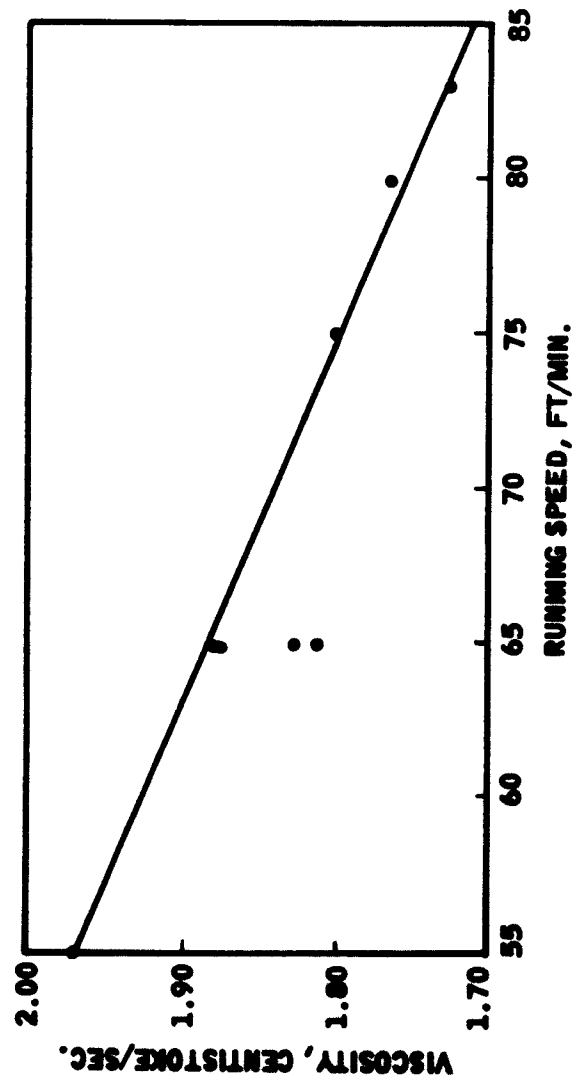
MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

PROCESS CONDITIONS: RUNNING SPEED AT 65 FT/MIN.
RESIN SPECIFIC GRAVITY OF 0.853 AT 75°F
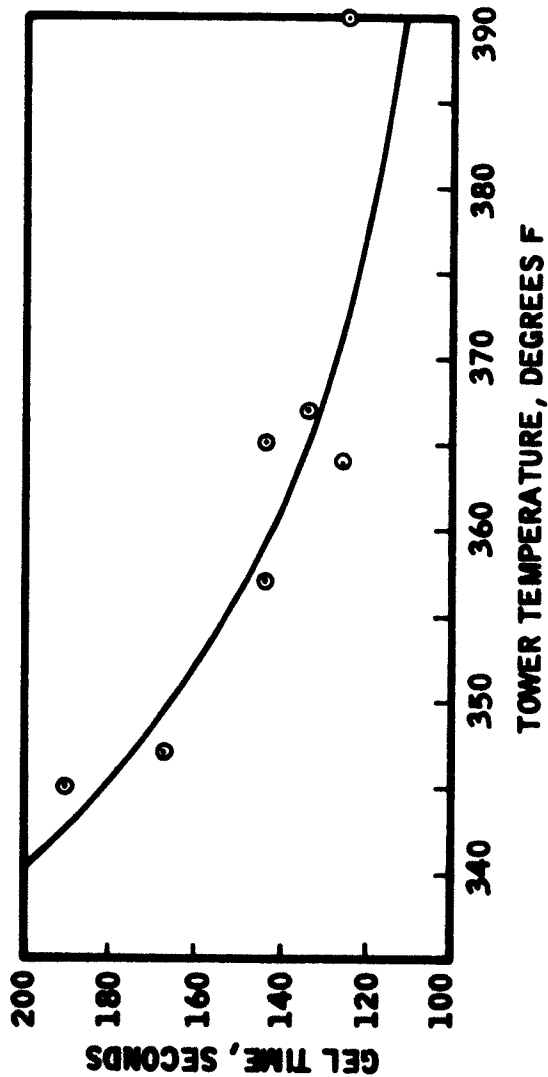
EFFECT OF TOWER TEMPERATURE ON GEL TIME



Figure 25

A270:63-899

MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

PROCESS CONDITIONS: TOWER TEMPERATURE AT 365° F
RESIN SPECIFIC GRAVITY OF 0.853 AT 75° F
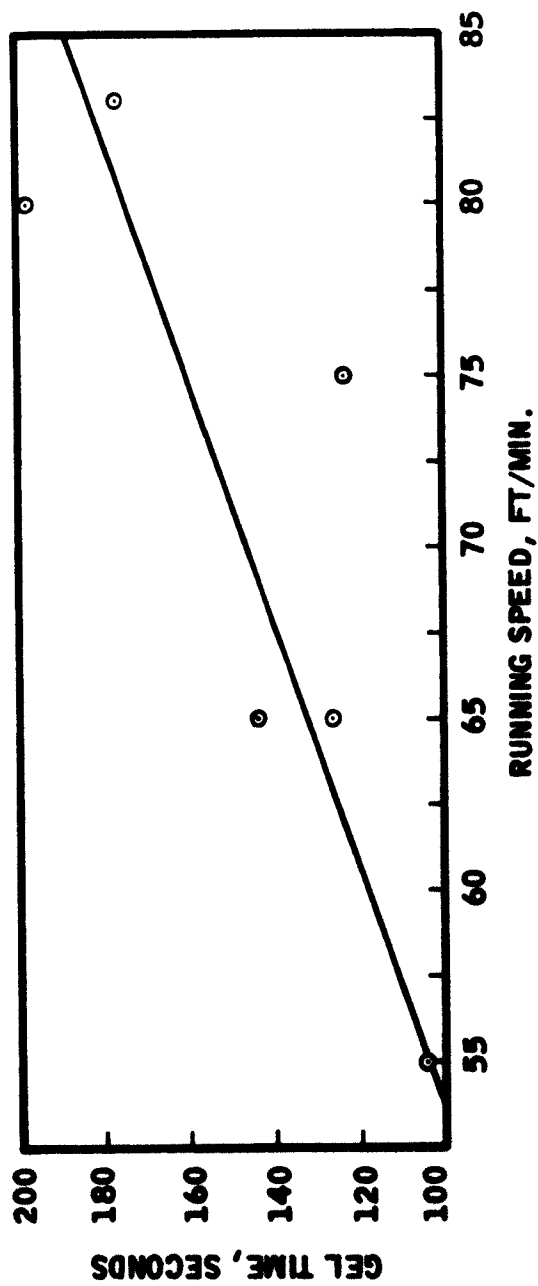


EFFECT OF RUNNING SPEED ON GEL TIME

Figure 26
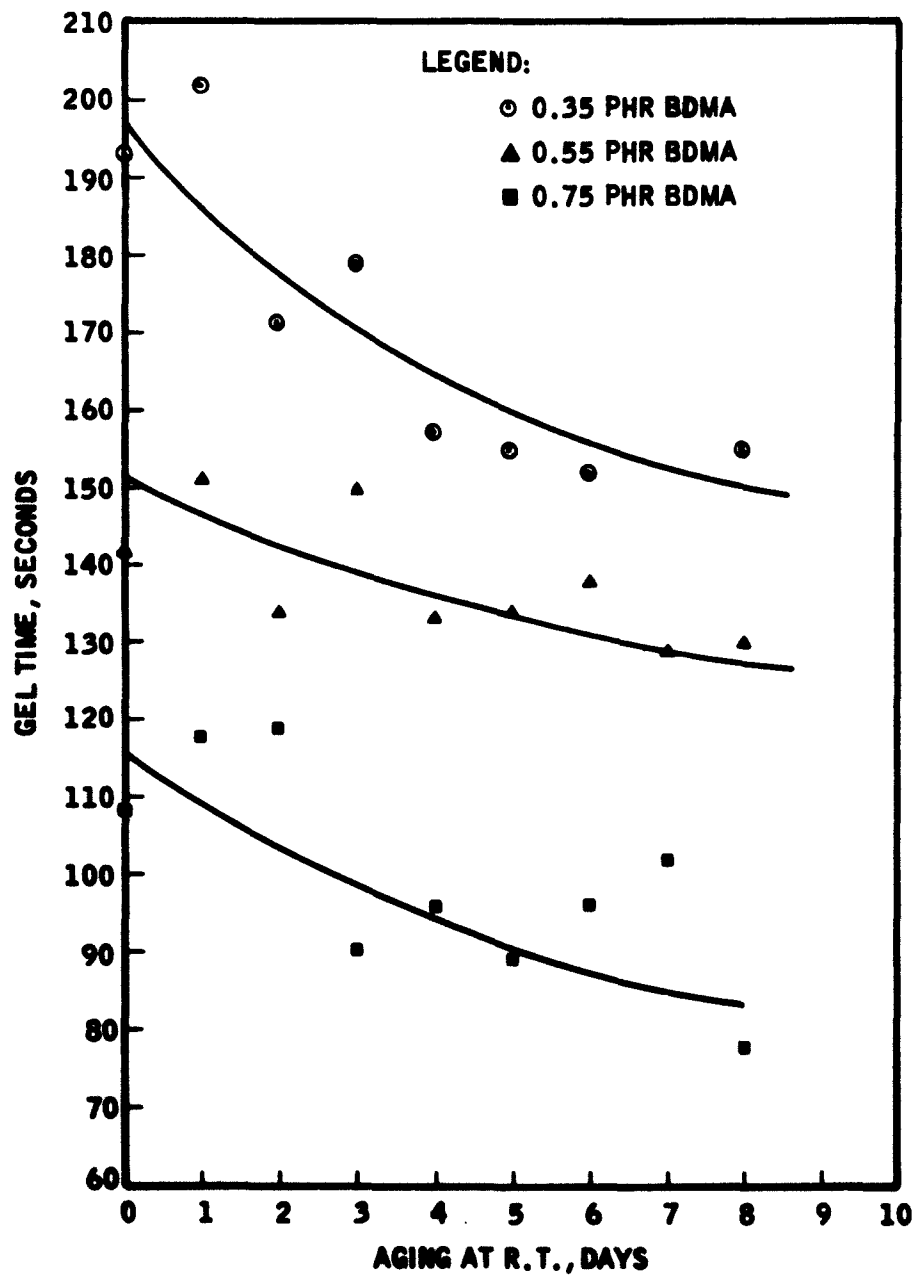
EFFECT OF AGING ON GEL TIME OF PREPREGS
CONTAINING VARIOUS AMOUNTS OF BDMA

A270:63-897

Figure 27

LEGEND:
- ⊙ RUNNING SPEED AT 80 FT/MIN.
- ■ RUNNING SPEED AT 75 FT/MIN
- ▲ TOWER TEMPERATURE AT 345°F
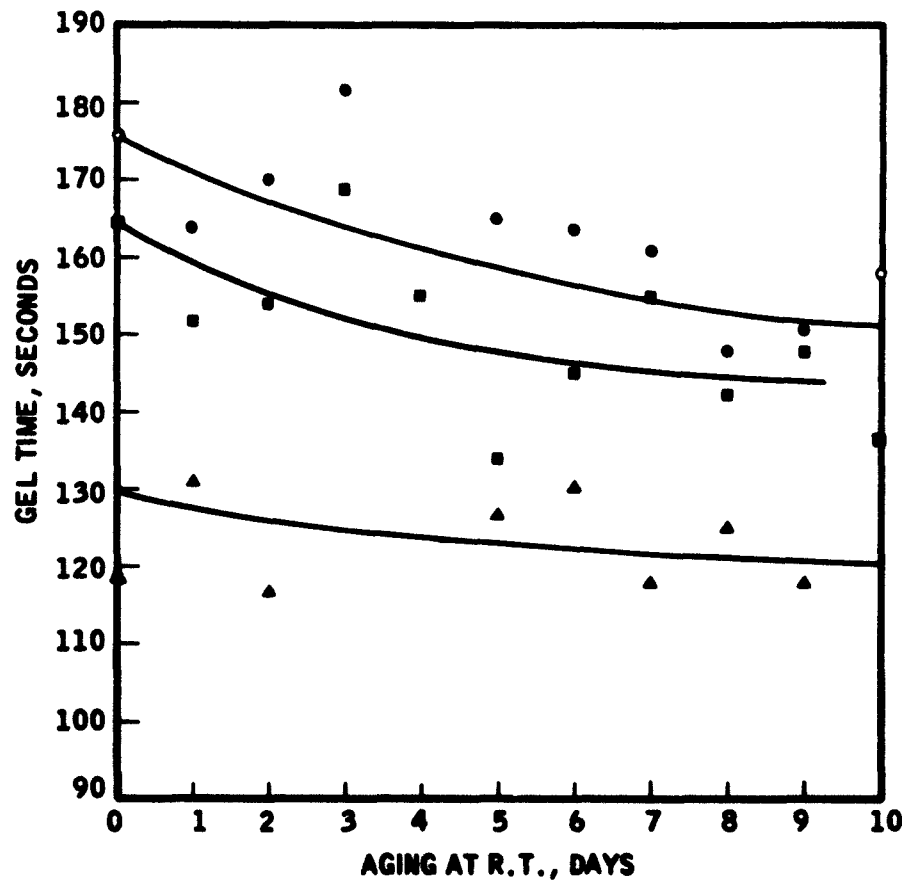


EFFECT OF AGING ON GEL TIME FOR PREPREGS

PRODUCED UNDER VARIOUS PROCESSING CONDITIONS

A270:63-897
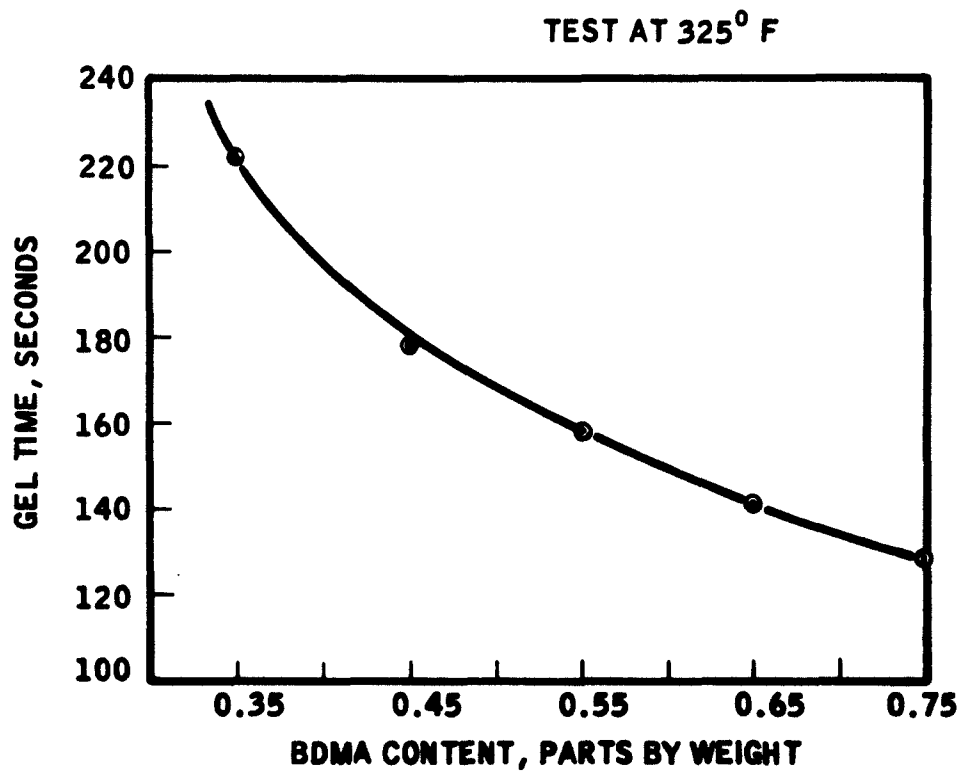
Figure 28

GEL TIME OF 58-68R RESIN SYSTEM WITH VARIOUS

AMOUNTS OF BDMA

A270:63-900
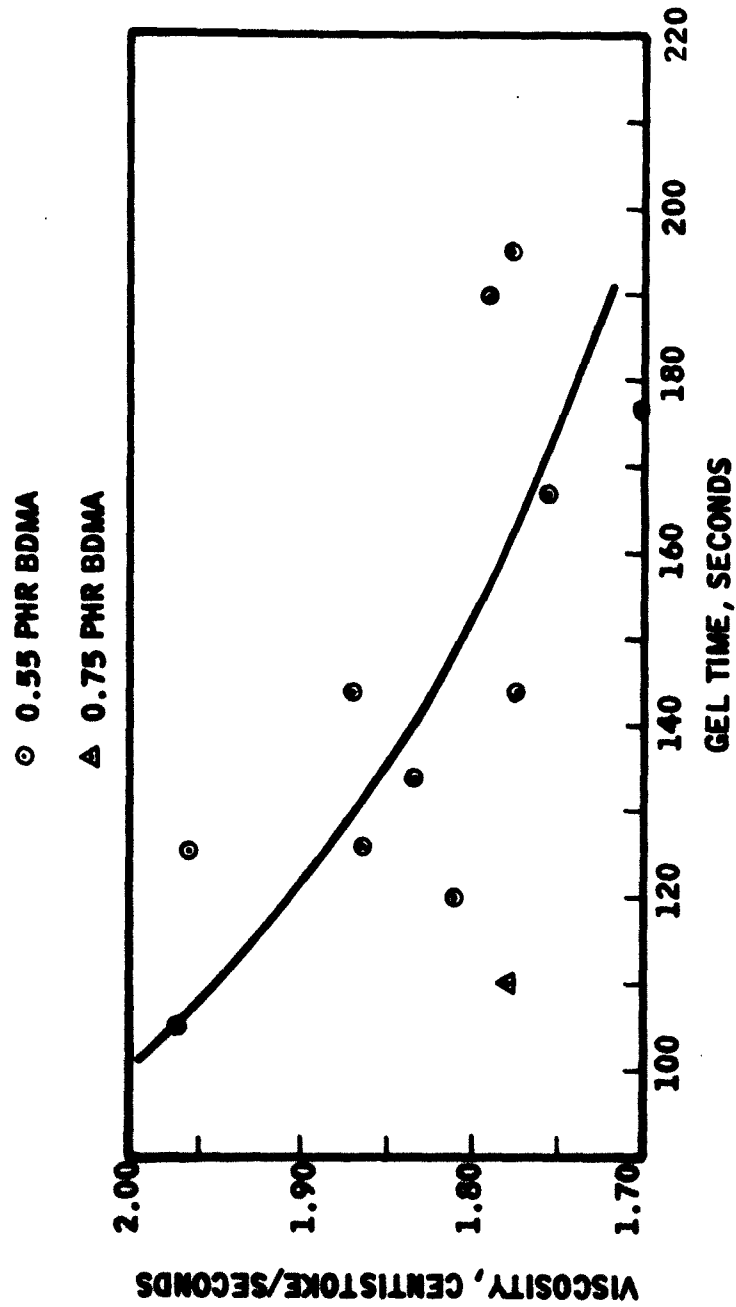
Figure 29

A270:63-902



CORRELATION OF GEL TIME AND VISCOSITY

Figure 30

## APPENDIX A

### PROCEDURE FOR THE DETERMINATION OF VISCOSITY

The equipment for the determination of kinematic viscosity includes a refractometer, calibrated Type No. 100 Canon-Fenske viscometer, graduated cylinders, constant-temperature water bath, and a stop watch. The resin from 50 to 60 yards of prepreg material is extracted with approximately 50 ml of DMF (dimethyl formamide; refractive index of $1.4300 \pm 0.0005$) and the solution filtered through Grade 512 filter paper. A 15-ml quantity of the filtrate is then standardized with DMF to refractive index of $1.4480 \pm 0.0002$ (19.4% solution by weight of resin). For standardization purposes, 1 ml of DMF reduces approximately 0.0014 unit refractive index. A 12-ml quantity of the standardized solution is transfered to the Canon-Fenske viscometer, and the temperature of the solution stabilized at $25° \pm 0.2°C$ in a constant-temperature water bath. (Passing the solution 3 times through the viscometer is normally satisfactory). Using a stop watch, time required to flow measured quantity of solution through the capillary tube of the viscometer is determined. An average efflux time is determined on three measurements taken for each solution with a maximum spread in measurements of 0.5 sec for the three readings. Efflux time is converted to viscosity in centistokes/sec by multiplving the time by a constant factor of the calibrated viscometer.

## APPENDIX B

### PROCEDURE FOR THE DETERMINATION OF GEL TIME

The equipment for the determination of gel time consists of the Fisher-Johns melting-point apparatus, glass cover discs, and a stop watch. A small segment of prepreg roving, approximately 1/8-in. long, is cut and placed between the glass cover discs. This specimen is placed on the melting-point apparatus, which is preheated to a constant temperature of 325°F (163°C). While heating, the resin is probed until gelation has occurred as observed through a magnifying glass. The gelatin or the end-point is reached when the resin viscosity is increased to a point where resin adheres to the glass surface and forms a mass of globules as contrasted to a clear, transparent liquid for the ungelled specimen. The gel time is that period of time between the placement of a specimen on the heated plate and the formation of a resin gel.

## APPENDIX C

### PROCEDURE FOR THE BAND WIDTH MEASUREMENT

For band width measurement with no pressure applied, prepreg roving un-spooled directly from a package is laid flat on a table and measurements taken to the nearest 0.001 in. using a micrometer caliper. For band width measurement with pressure applied, a prepreg roving 6-ft long is tied at one end to a fixed pin or a holder, and a 10-lb weight is attached to the other end. The weighted end is then suspended over the edge of the table, which is measured 4 ft from the pin. Three band width measurements are taken along the length of the speci-men at 1-ft increments from the pin. Areas of sudden band width change, such as those occurring at the traverse termination point of a waywind package, are avoided for the measurement. Another measurement is taken at the table edge, where the largest spread in band width occurs under tension.

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